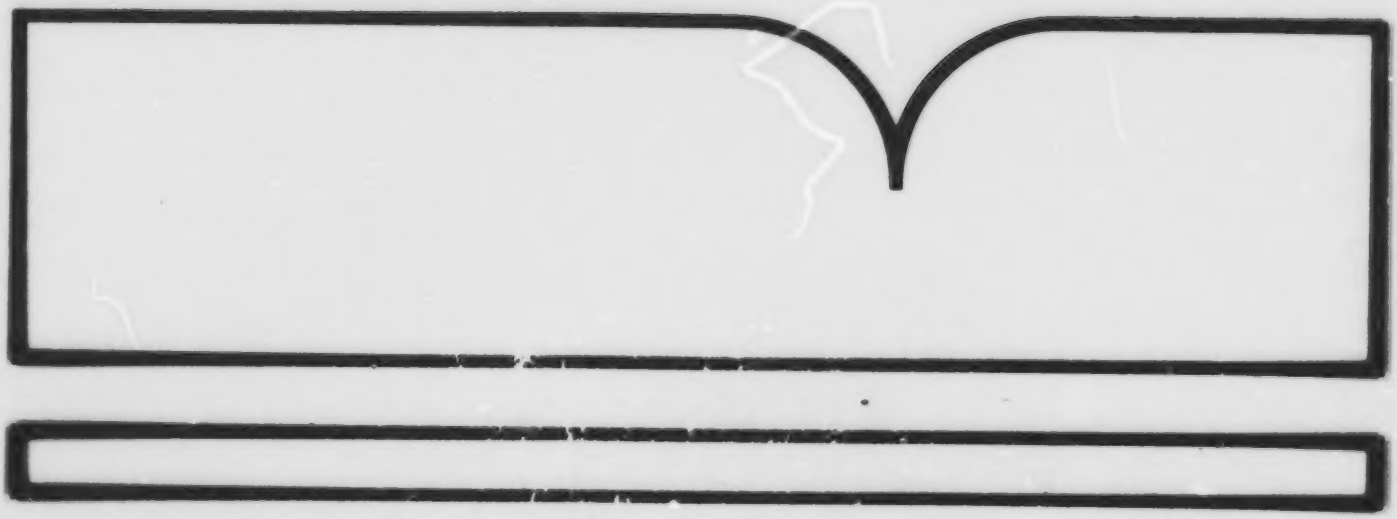
PB85-178424

Southeast Chicago Air Quality: A Plan for the Evaluation of PCBs (Polychlorinated biphenyls)

Illinois State Environmental Protection Agency
Springfield

Feb 85



U.S. Department of Commerce
National Technical Information Service
NTIS

Illinois
Environmental
Protection Agency

Division of Air Pollution Control
2200 Churchill Road
Springfield, Illinois 62706

February, 1985

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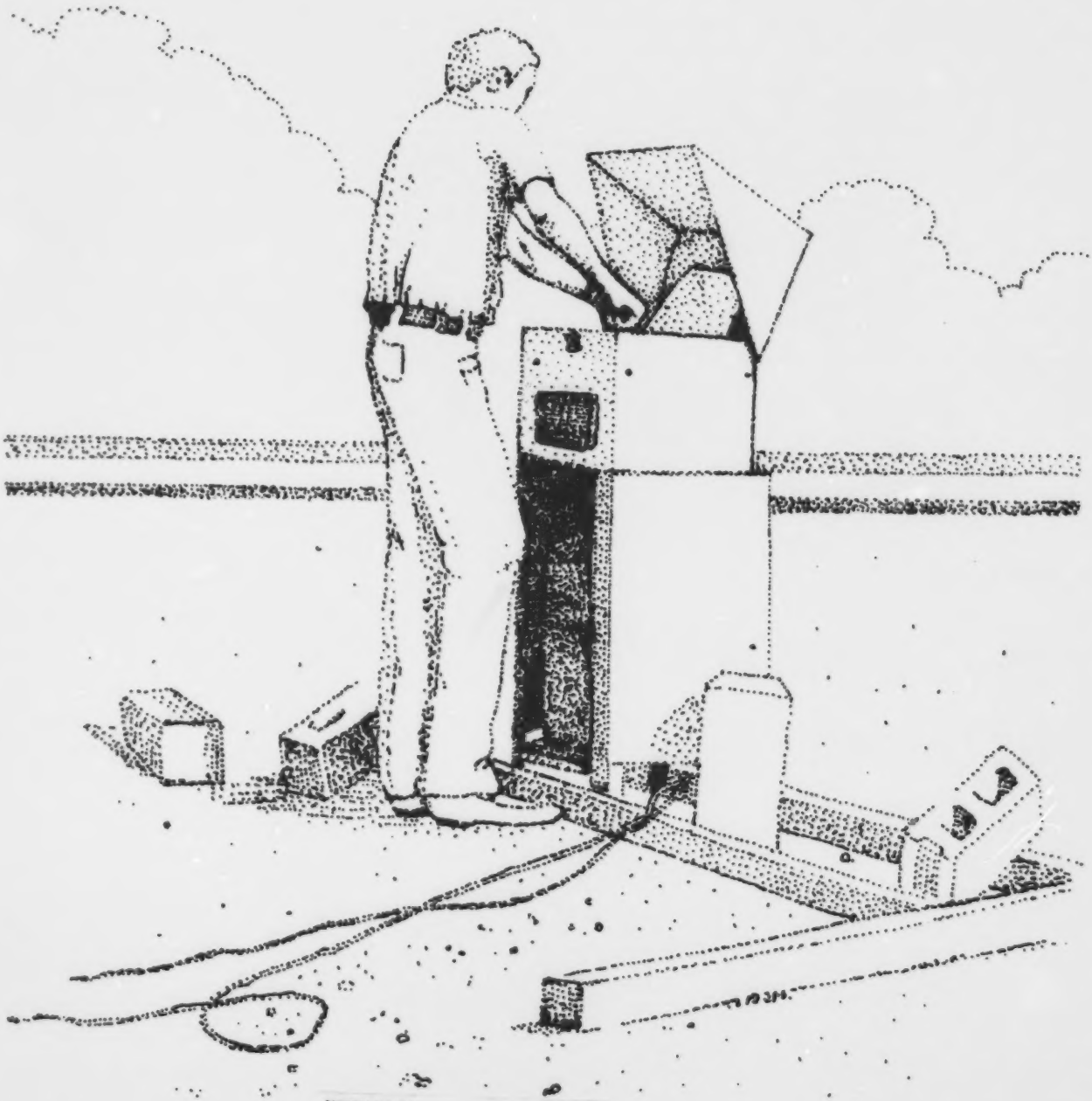
IEPA/APC/85-003

SOUTHEAST CHICAGO AIR QUALITY:

A Plan for the Evaluation of PCB's



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Project Plan for the Sampling and Analysis of Polychlorinated Biphenyls in the Lake Calumet Area of Southeast Chicago

February, 1985

Illinois Environmental Protection Agency
Ambient Air Monitoring Section
2200 Churchill Road
Springfield, Illinois 62706

APPROVALS

IEPA Program Manager David J. Kolaz DATE 2/19/85

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IEPA QA Officer James Buckert DATE 2/20/85

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1.0 PROJECT DESCRIPTION

1.1 Introduction

Polychlorinated biphenyls (PCBs) are highly toxic agents which have been shown to cause harmful affects in numerous animal species, including man.[1-3] PCBs have been used and manufactured in Europe, Japan, the Soviet Union, and the United States. The characteristics that cause PCBs to have broad industrial uses also make their safe disposal very difficult. The environmentally preferred option for disposing of PCB containing wastes is incineration. At the present time, three non-mobile incinerators have been approved by the USEPA for burning PCBs in the United States. One of the approved incinerators is located in Deer Park, Texas, another in Eldorado, Arkansas, and the third is the SCA Chemical Services incinerator located at 11700 South Stoney Island Avenue, in the Lake Calumet area of Chicago, Illinois. Based in large part on the results of stack tests from five trial burns conducted in October of 1982, the USEPA and Illinois EPA approved permits to incinerate liquid and non-liquid PCB wastes at the Chicago facility. The Illinois IEPA will be conducting a four month field monitoring program to sample and analyze PCBs in the atmosphere around the SCA incinerator to further assess the environmental impact of the incineration of PCBs. This document presents the overall plan for this project.

1.2 Objectives

The program plan has been developed in order to accomplish the following objectives:

1. Quantitatively determine PCB levels in the ambient air at two locations in the vicinity of the SCA incinerator in Southeast Chicago.

2. Correlate the air sampling results with the operational parameters from the SCA incinerator.

3. Evaluate the environmental risk due to the incineration of PCBs.

4. Use the information from this study to prepare a more comprehensive PCB sampling program.

1.3 Study Area Description

1.3.1 Geographic and Demographic Characteristics

The study area is located in the southeastern portion of Chicago near Lake Calumet. As shown in Figure 1.1, the study area is bounded on the north by 95th Street, on the South by Sibley Boulevard, on the east by the Illinois/Indiana border, and on the west by Martin Luther King, Jr. Drive and the Chicago & Western Indiana Railroad tracks.

The above boundaries describe an area of approximately 26 square miles with a population of approximately 110,000 people. According to the 1980 census[4], approximately 30% of the residents are less than 18 years of age and nearly 10% are 65 years of age or older.

A dominant feature of the Lake Calumet study area is the water resources. The study area includes Lake Calumet, Wolf Lake, the Calumet River, the Little Calumet River, and the Grand Calumet River. There are several areas of wet lands generally in the eastern portion of the study area. The low marshy areas have been used to dump an extensive amount of waste material.

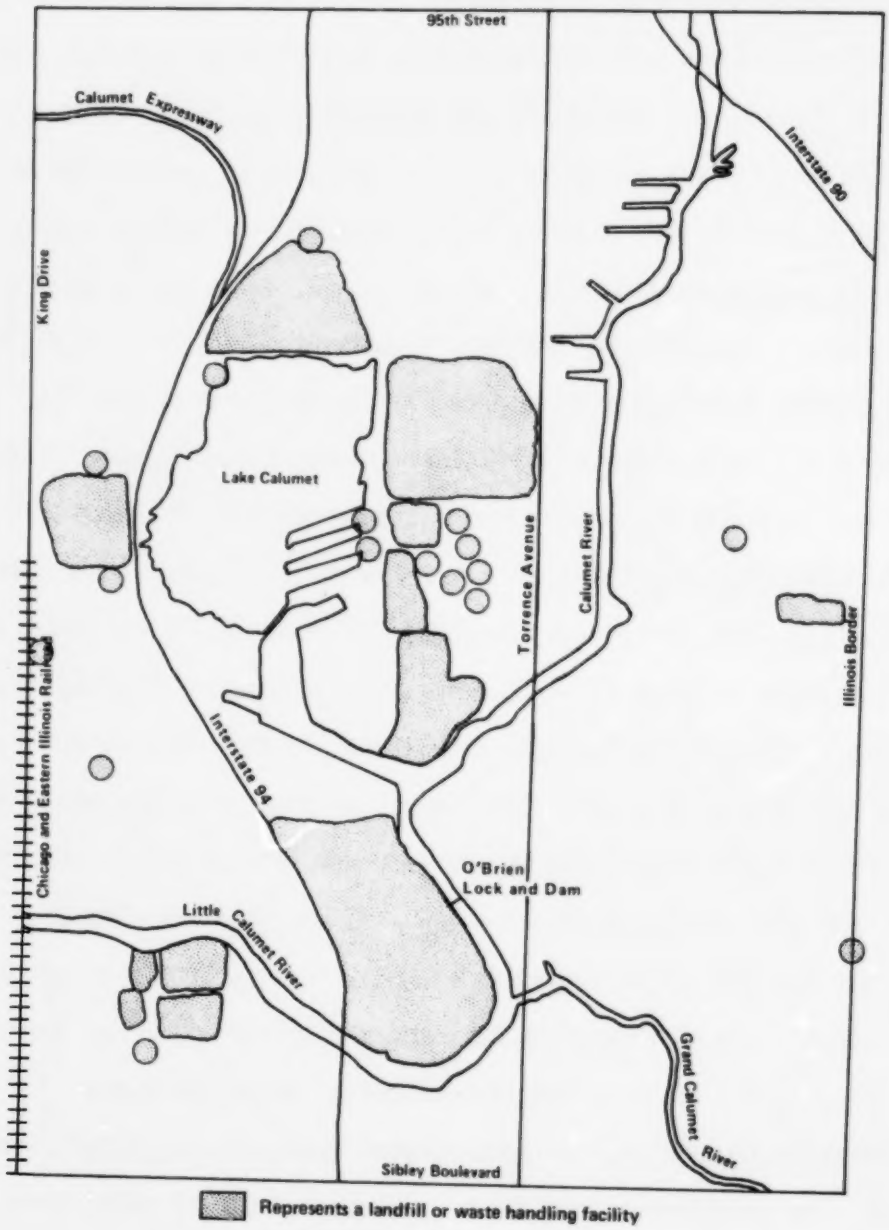

Figure 1.1 Lake Calumet Study Area

1.3.2 Emission Sources

The Lake Calumet area of southeast Chicago is a heavily industrialized region with a long history as a disposal area for a wide variety of industrial, commercial and residential wastes.[5] There are 22 major facilities which emit over 100 tons per year of particulate matter, sulfur dioxide, nitrogen dioxide, carbon monoxide, or hydrocarbons. These facilities include steel mills, chemical plants, and assembly plants. This project is specifically directed to measuring ambient levels of PCBs near the known major potential source of PCBs in the area; the SCA Chemical Waste incinerator at 11700 South Stoney Island Avenue. This incinerator consists of a refractory lined rotary kiln followed by a liquid fired refractory lined secondary combustion chamber, two packed scrubbers and four ionizing wet scrubbers. The maximum capacity of the combined system is 120 million Btu/hr. In addition, the liquid PCB waste feed rate is limited to less than 6012 lb/hr and the non-liquid PCB waste feed to less than 2910 lb/hr. The combustion gases from the rotary kiln enter the secondary combustion chamber for complete oxidation. Stack tests[6] from this incinerator have shown a destruction and removal efficiency for PCBs exceeding 99.9999% while burning liquid or solid PCB laden wastes. These stack tests have shown that extremely low, but measurable, levels of PCBs, trichlorobenzenes, polychlorinated dibenzodioxins (PCDD's) and polychlorinated dibenzofurans (PCDF's) were emitted. The trichlorobenzene makes up a portion of the waste feed, while the PCDD's and PCDF's are combustion products of the incineration process. The results of the PCB stack tests are summarized in Appendix A.

1.4 Monitoring Program Plan

The IEPA will be conducting a four month field monitoring program during the period November 1984 to March, 1985 for the purposes of measuring PCB levels in the ambient air in the vicinity of the SCA Chemical Waste incinerator. Samples will be collected using a device (General Metal Works Model PS-1 PUF Sampler) capable of quantitatively retaining particulates and organic vapors.[7,8] The material used to collect the samples to be tested will consist of polyurethane foam (PUF) between which is placed a solid adsorbent (Florosil). The collection efficiency of the system for PCBs is expected to exceed 85%.[7-10] Further information on the sampling system is presented in Section 3. The collected samples will be analyzed for PCBs after the sample is extracted using a 5% ether/hexane solvent. The PCBs will be analyzed as Aroclor 1242, by EPA Method 608 (Gas Chromotography with Electron Capture Detection).[11] Additional information on the analytical procedures is provided in Section 4 and Appendix E.

Appropriate quality assurance measures will be taken to ensure the data is of known precision and accuracy. The program will include analysis of field blanks, laboratory blanks, performance audits, and the collection and retention efficiency of PCB spiked samples.

1.4.1 Sampling Locations

Samples will be collected at two locations; one northeast of the SCA incinerator and one southeast. The northeast site is located at Orville T. Bright Elementary School, 10740 South Calhoun. This sampling location is 2.3 kilometers from the SCA incinerator and is downwind when the wind flow is out of the southwest at 212 degrees. The Virgil S. Grissom Elementary School, 12810 South Escanaba, is southeast of the incinerator at a distance of 3.0 kilometers. Winds from the northeast at 319 degrees have the potential of bringing incinerator emissions to this site. Both of these sites are shown in Figure 1.2 along with the location of the incinerator. PCB samples are taken when a site is downwind from the incinerator for 12 hours or more during periods of time when the incinerator is burning PCBs. Dispersion modelling results (Appendix B) have shown that detectable PCB concentrations at the sampling locations are unlikely to occur with the incinerator operating according to approved permit conditions.

1.5 Project Schedule

The air sampling program began about November 1, 1984 and will continue through March 1, 1985 or until 16 samples have been collected. A final report will be completed within 90 days of the completion of the project and will contain all data, quality assurance reports, and data analysis.

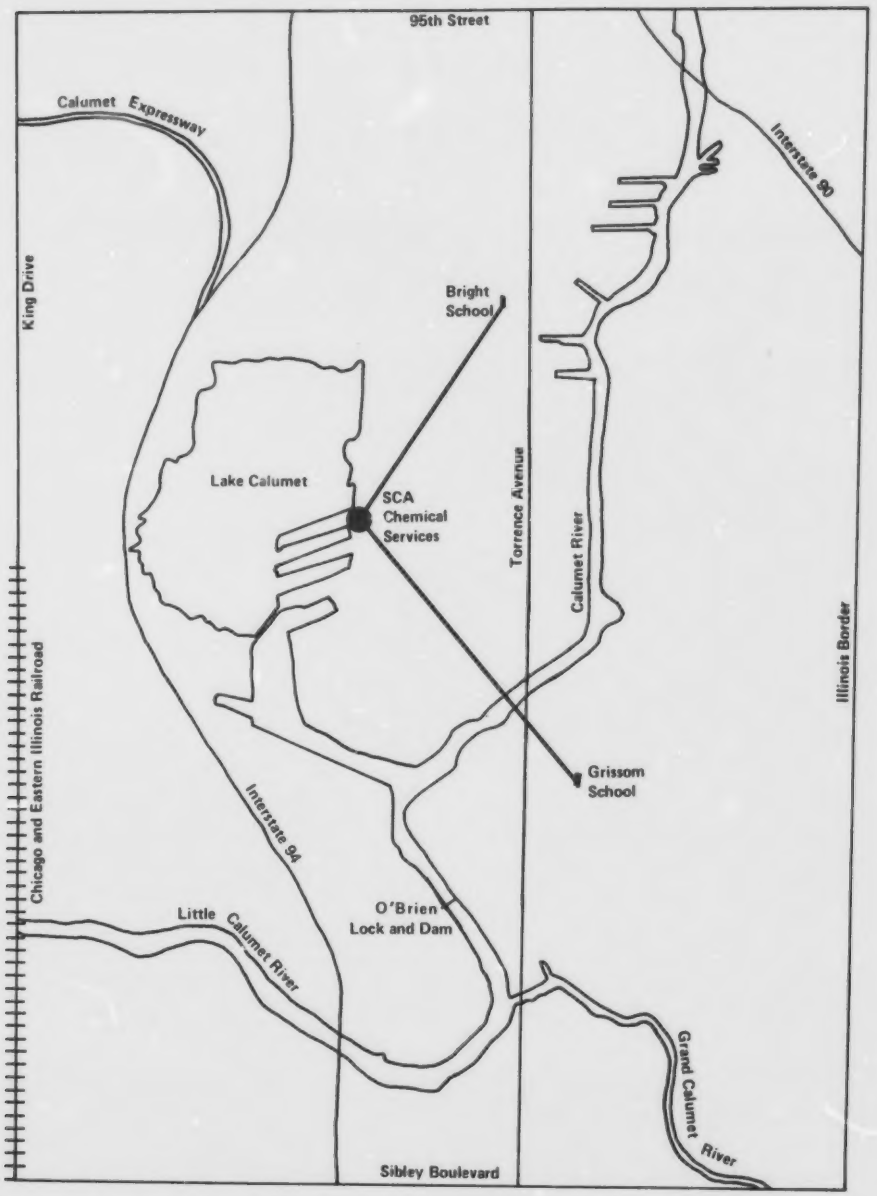

Figure 1.2 PCB Sampling Locations

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

The Illinois Environmental Protection Agency (Illinois EPA) will be conducting this program through its Division of Air Pollution Control, Ambient Air Monitoring Section. Mr. David Kolaz will serve as Program Manager for this study. Mr. Kolaz will report directly to Mr. Michael P. Hayes, Acting Manager of the Division of Air Pollution Control. Management personnel responsible for this program will be: Mr. Robert E. Hutton, Manager of the Field Operation Section, Mr. Terry A. Sweitzer, Manager of the Data Management Section, and Mr. James Buckert, Quality Assurance Officer, and Senior Technical Advisor.

Mr. Kolaz will have prime responsibility for organizing the project team and managing the team to successfully complete this study on schedule. Figure 2.1 shows the project organization for this task. The Project Director will be Mr. Robert E. Hutton, who will be responsible for the technical direction of the study. The field team will be comprised of two Illinois EPA staff members who will conduct the sample collection. Mr. Thomas Barounis and Mr. John Abate will have the responsibility of collecting ambient air samples according to the approved protocol. The analysis, validation, and reporting of the data from this program will be the responsibility of Mr. Terry A. Sweitzer, Mr. Robert Swinford, and Mrs. Pamela Henricks.

The Radian Corporation, under a contract to the Illinois EPA, will be responsible for supplying clean sampling cartridges for the PCB Field

Monitoring Program, analyzing the cartridges for PCBs after sampling, and reporting all analytical data (including samples, blanks, and spikes) to the Illinois EPA. Ms. Maureen McDevitt will be Radian's project coordinator for this project.

The Quality Assurance Project Officer for this study will be Mr. James Buckert who will also serve as Senior Technical Advisor. In his capacity as Quality Assurance Officer, Mr. Buckert will be responsible for surveillance and formal reporting of all quality assurance program activities to ensure data are collected which meet the accuracy, precision, completeness, representativeness, and comparability objectives set forth in this air monitoring program plan. Mr. Buckert will report directly to Mr. Kolaz, the Program Manager, on quality assurance matters. As Senior Technical Advisor, Mr. Buckert will provide technical support relevant to all areas of the programs and will interact directly with the Radian Corporation. All technical support will be provided through Mr. Hutton, the Project Director.

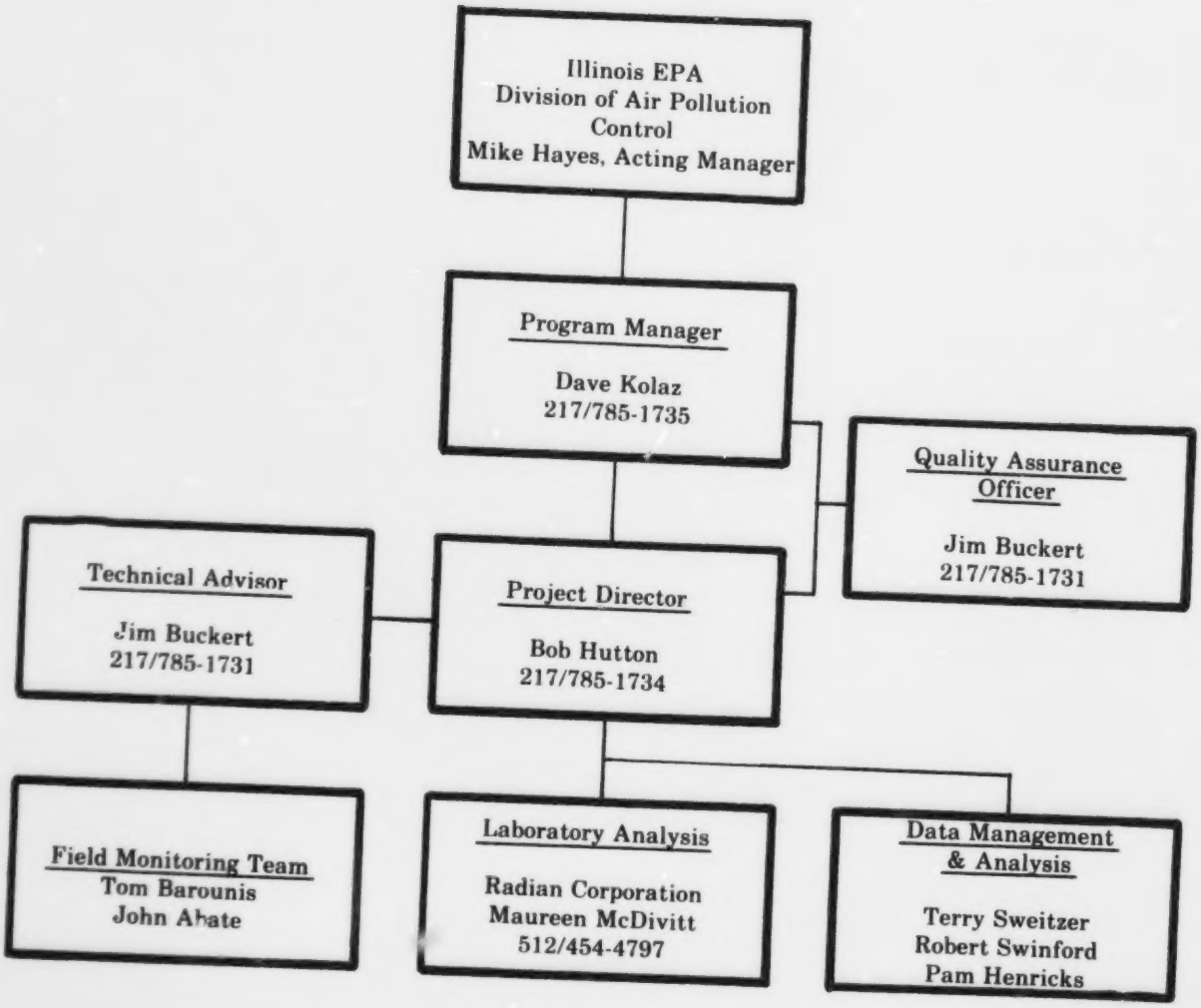

Figure 2.1 PCB Project Organization

3.0 SAMPLING PROCEDURES

3.1 Monitoring Equipment

PCB field monitoring will be performed using General Metals Works, Inc. (GMW) Model PS-1 air samplers. Schematics of the sampler and its components are presented in Figures 3.1 and 3.2. The sampling system is housed in a standard high volume shelter and contains a special sampling inlet (i.e., sampling module), a venturi-magnehelic gauge assembly, 7 day Paragon timer, elapsed time meter, voltage controller, ball-valve, motor, and exhaust hose. Power requirements are 115 volts AC at 60 hertz.

The aluminum sampling module is designed to hold a 100 mm diameter filter at its inlet and a glass sampling cartridge in its lower canister. The circular filters are cut from 8" x 10" glass fiber filters supplied by the USEPA for the 1984 TSP sampling year (filter lot and batch numbers 34373 CP). The sampling cartridge is a 60 mm i.d. (63.5 mm o.d.) x 130 mm long borosilicate glass cylinder. A narrow indentation encircles the cylinder approximately 25 mm from one end and holds a wire mesh support screen in place. The cartridge is loaded with 25 ml of 16/30 mesh, PR grade Florisil sorbent sandwiched between two polyurethane foam (PUF) plugs. The top PUF plug is approximately 50 mm long, while the bottom plug is only 25 mm in length. Both plugs are approximately 65 mm in diameter and have densities near 0.02 g/cm3. The metal screen provides a base to support the PUF plugs and sorbent. A loaded cartridge is displayed in Figure 3.3.

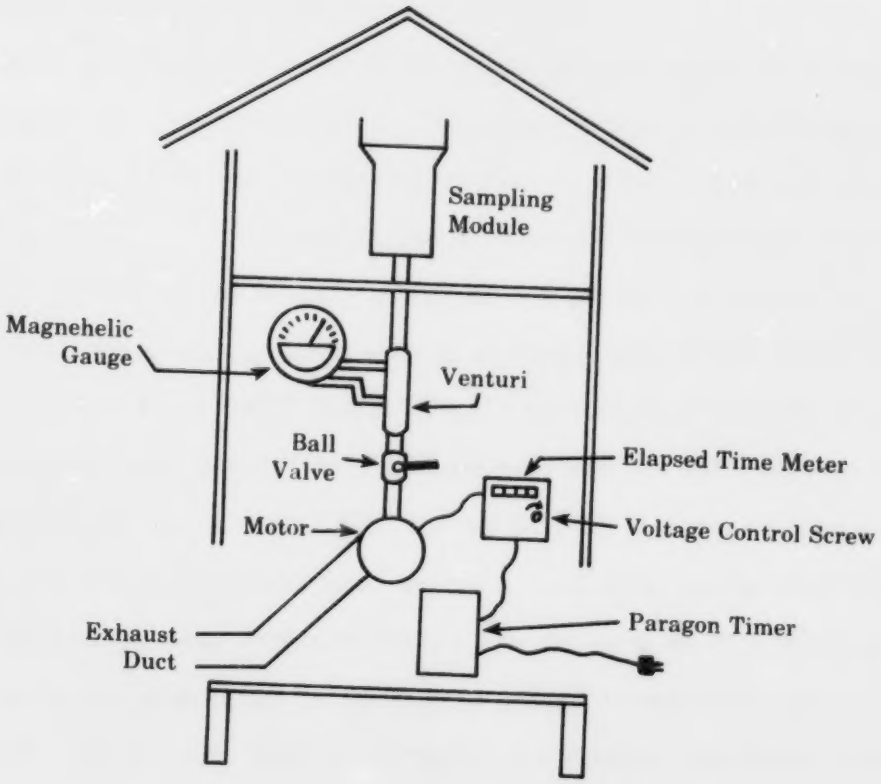

Figure 3.1 PCB Sampler

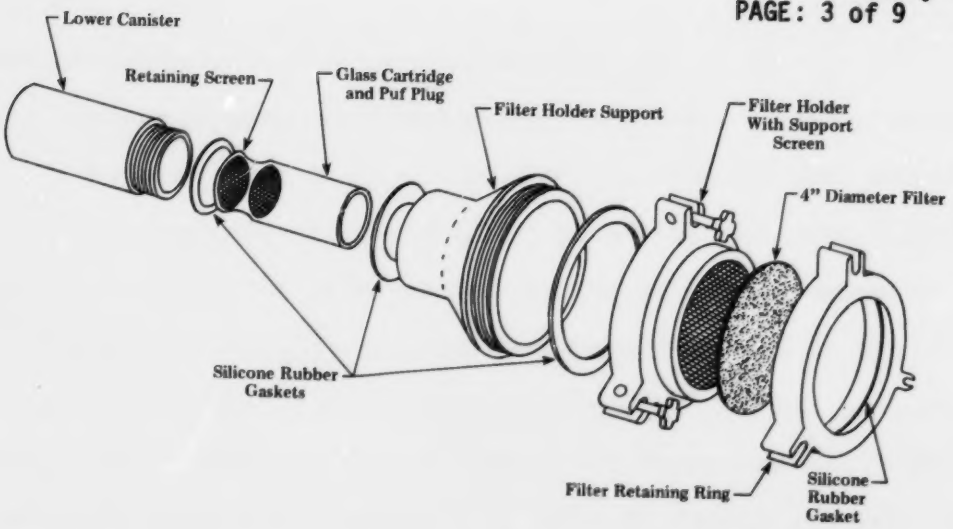

Figure 3.2 PCB Sampler Components

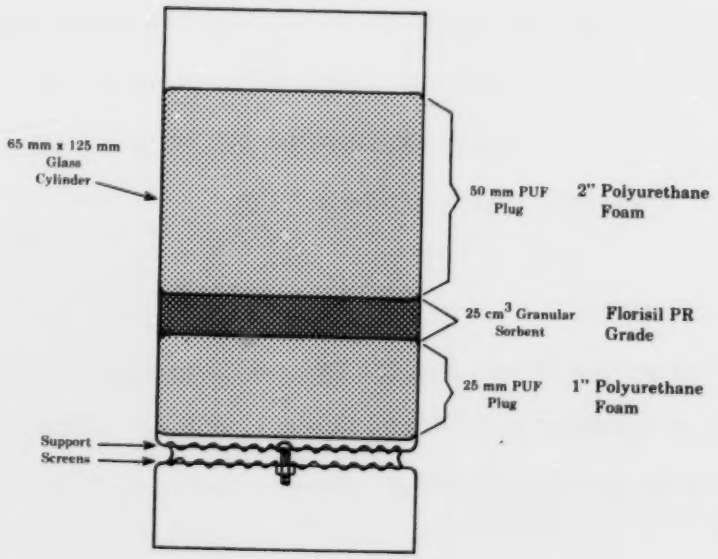

Figure 3.3 PCB Sampling Cartridge

Air flow through the sampler is measured by an in-line venturi-magnehelic gauge assembly located immediately downstream of the sampling module. At the start of each sampling event the flow rate is set to approximately 8 theoretical12 cubic feet per minute (tcfm) by adjusting the sampler's ball-valve assembly until the magnehelic gauge is at the appropriate value. The ball-valve is situated between the venturi and the sampler's motor.

Activation and deactivation of sampling can be accomplished manually or by presetting a 7-day Paragon timer. When the Paragon timer is used, start and stop times are accurate to within $\pm$ 15 minutes. An elapsed time meter, electrically connected between the Paragon timer and sampler motor is used to measure total sampling times. This meter records time to the nearest 0.1 minute and is accurate to within $\pm$ 2 minutes over a 24 hour period. Also located with the elapsed time meter is an adjustable voltage controller which can be used to set the motor voltage to any value between 0 and line VAC. An exhaust hose, six feet in length, is attached to the motor and is used to direct the motor's exhaust away from the sampler.

3.2 Monitoring Strategy

PCB sampling at the Bright and the Grissom Elementary Schools will be performed between November 1, 1984 and March 1, 1985 on a non-scheduled basis. During this period a total of sixteen samples will be collected

over a 24 ± 1 hour duration and analyzed for PCBs. Initiation of each sampling event will be determined by the Project Director using current meteorological projections from the National Weather Service and operational information from the SCA incineration facility. Sampling may begin at Bright Elementary School if the forecasted wind directions are between 190 and 235o for 12 hours or more. Projected wind directions between 300 and 340o for at least 12 hours are required for sampling to begin at Grissom Elementary School. Sampling at either site is generally prohibited if the SCA incinerator is down or if meteorological projections indicate heavy and protracted precipitation. Exceptions to these conditions may be made by the Program Manager.

3.3 Standard Procedures for the GMW Model PS-1 Air Sampler

3.3.1 Calibration and Audit of the GMW PS-1 Sampler

Calibrations and audits of the GMW PS-1 sampler are performed by a designated field operator using a GMW Model 40 orifice calibration unit (OCU). This unit is initially calibrated by the manufacturer against a standard rootsmeter (see Appendix C for the most recent calibration data). The OCU is recalibrated annually by returning it to the manufacturer. The manufacturer's calibration is accepted only if a linear regression analysis of the data yields a correlation coefficient of at least .9998.

Each GMW PS-1 sampler is flow calibrated upon installation at a designated site (see Appendix D, SOP FO-050). The samplers are then recalibrated every six months, upon replacement of a venturi or magnehelic gauge, or when a one point flow rate audit near 8 tcfm exceeds ±10% difference. The calibration information is recorded on a standardized data form (see Appendix D SOP FO-050) which, when completed, is forwarded to the Data Management and Analysis Unit (DMAU) in Springfield.

An initial review of the calibration data is made by the DMAU using specific criteria, as documented in SOP DMA-027 (see Appendix D). If the information is complete, then a calibration table with the sampler's magnehelic gauge readings and corresponding flow rates is generated. The assumed relationship between these two values is;

$$Y = m X^{.5} + b$$

where, X = magnehelic gauge reading, in inches water

Y = corresponding flow rate, in tcfm

m = linear regression slope

b = linear regression intercept

as long as the resulting correlation coefficient is at least .9990. If information on the field generated data form is not complete, or if the regression correlation coefficient derived from the calibration data is less than .9990, then the calibration is void and must be redone.

Flow rate audits are performed just prior to every third sampling event on a given sampler and are used to check the sampler's dynamic operation. The audits are conducted in a manner almost identical to that presented in SOP FO-050, except that only one flow rate near 8 tcfm is measured. This flow rate is obtained by adjusting the sampler's ball-valve to a magnehelic gauge reading of 40 inches H_2O. For audit calculations, the flow rate measured through the OCU is designated as the X variable. The sampler's flow rate is derived from the most recent calibration table using the measured magnehelic gauge reading. The derived sampler flow is specified as the Y variable and the percent difference in flow rates is calculated from the following equation:

$$\frac{(Y-X)}{X} * 100 = \% \text{ difference } (D_i)$$

All audit data are entered under "Comments" on the PS-1 Sample Information Form (see Appendix D, SOP FO-051).

3.3.2 Operation of the GMW Model PS-1 Sampler

A detailed procedure on the operation of the PS-1 sampler is provided in Appendix D, SOP FO-051.

3.4 Sample Cartridge and Filter Handling and Storage

Near the beginning of the project, the Radian Corporation in Austin, Texas will ship ten clean sampling cartridges and thirty-two 100 mm diameter glass fiber filters to the Illinois EPA field office in Maywood, Illinois. The cartridges will contain the requisite PUF and Florosil sorbent, be wrapped in n-hexane rinsed aluminum foil, and be sealed in individual glass jar containers. The fiber filters will be sealed in separate aluminum pouches. At the Maywood office, these materials will be stored at room temperature, in a clean and secure area, until needed for sampling.

On the day of sampling, two sampling cartridges in their glass containers and two filters in their pouches are selected and placed in an uncharged ice chest for transport to the sampling location. In order to minimize shelf life, cartridges which have been retained at Maywood the longest will be chosen first. Of those selected, one cartridge and filter is specified as the trip blank, while the remaining cartridge and filter are installed in the PS-1 sampler. At the end of sampling, the sample cartridge and filter are separately rewrapped in their aluminum foil and placed in the appropriate cartridge container. The trip blank cartridge and filter are also rewrapped and returned to the appropriate

glass container. Both containers are labelled and then placed in an ice chest, which is maintained at -6oC with charged ice packs. The ice chest is returned to Maywood, where the two cartridge containers and ice packs are transferred to an insulated shipping container. The shipping container is sent to Radian via express mail no later than twenty-four hours after the end of the sampling period.

4.0 ANALYTICAL PROCEDURES

At the end of the monitoring project the Radian Corporation will submit all relevant analytical standard operating procedures used by them during the course of the project. As a consequence, reference to specific analytical procedures are not made in this section of the monitoring plan. However, the following paragraphs provide an outline of the PCB analytical protocol to be used by Radian.

4.1 Initial Cartridge Cleanup and Filter Preparation

The IEPA will supply Radian with ten sampling cartridges containing the requisite PUF and Florosil sorbent, a sufficient quantity of glass jars to be used as containers for the sampling cartridges, and thirty-eight 100 mm diameter glass fiber filters. Each cartridge will be cleaned in a 500 ml Soxhlet extractor by extracting with 200 to 300 ml of acetone for 14 to 24 hours at approximately 4 cycles per hour. Following extraction, the extracts will be discarded and the cartridges dried in a vacuum oven at room temperature.

Of the ten cartridges cleaned, two will be re-extracted in 500 ml Soxhlets with 200-300 ml of 5% diethyl ether in n-hexane (v/v) for 14 to 24 hours at approximately 4 cycles per hour. Each extract will be concentrated and analyzed for PCBs as Aroclor 1242. If either cartridge contains more than 50 nanograms PCB, then all ten cartridges must be re-extracted until the limit is met.

The dried cartridges are to be wrapped in n-hexane rinsed aluminum foil and placed in the cleaned glass jar containers. These loaded containers will be stored in a clean area at Radian until required by the Illinois EPA.

Six filters from the group of thirty-eight will be randomly selected for blank analysis. The six filters will be grouped together in one Soxhlet extractor and extracted with 5% diethyl ether in n-hexane (v/v) for 14 to 24 hours at approximately 4 cycles per hour. After extraction the filters are discarded, but the extract is concentrated and analyzed for PCBs as Aroclor 1242. If the total extract contains more than 50 nanograms PCB, then the Senior Technical Advisor will be notified and the problem resolved by the Project Director at that time. If the limit is met, then the remaining thirty-two filters will be placed in separate n-hexane rinsed aluminum pouches and stored in a clean area until needed by the Illinois EPA.

4.2 Spike Recovery and Carry-over Test

Prior to field sampling Radian will perform a spike recovery and carry-over test on the cleaned sampling cartridges at two different PCB levels. To one cartridge, approximately .12 micrograms of Aroclor 1242 will be injected under the surface of the upper PUF plug. An injection of .24 micrograms of the Aroclor will be made into a second cartridge.

Both spiked cartridges will then be individually extracted, and the extracts concentrated and analyzed. Both samples will be analyzed in duplicate. From the measured amounts of Aroclor extracted, an average percent difference for the replicate analyses and percent analytical efficiencies can be calculated using the following equations:

X_{11} = measured amount of Aroclor in the extract obtained from the .12 ug injection, in ug.

X_{12} = replicate measurement of the extract obtained from the .12 ug injection, in ug.

A_1 = average percent difference of the replicate measurements obtained from the .12 ug injection.

$$A_1 = \frac{X_{11} - X_{12}}{(X_{11} + X_{12})} * 200$$

X_{21} = measured amount of Aroclor in the extract obtained from the .24 ug injection, in ug.

X_{22} = replicate measurement of the extract obtained from the .24 ug injection, in ug.

A_2 = average percent difference of the replicate measurements obtained from the .24 ug injection.

$$A_2 = \frac{X_{21} - X_{22}}{(X_{21} + X_{22})} * 200$$

Y_1 = known amount of Aroclor injected into the first cartridge (approx. 12 ug), in ug.

Y_2 = known amount of Aroclor injected into the second cartridge (approx. .24 ug), in ug.

E_1 = percent analytical efficiency for the .12 ug range.

$$E_1 = \frac{X_{11} + X_{12}}{Y_1} * 50$$

E_2 = percent analytical efficiency for the .24 ug range.

$$E_2 = \frac{X_{21} + X_{22}}{Y_2} * 50$$

No quality control limits are established for the average percent differences between the replicate analyses or for the percent analytical efficiencies. It is estimated that the replicates will be within 10% of their average and the analytical efficiencies will be between 120% and 70%. For this project, the spike test is performed only to generate information which may be useful for quality control in a future project.

After the spike extractions, the two cartridges will be tested for carry-over by immediately re-extracting, and then concentrating and analyzing the extracts. If either extract contains more than 50 nanograms PCB, then the Senior Technical Advisor will be notified and an appropriate course of action determined by the Project Director.

4.3 Sample Extraction, Concentration, and Analysis

When an insulated shipping container is received from the Illinois EPA, the two jars with the sample cartridge-filter pair and trip blank cartridge-filter pair are removed and stored at 5oC or less until needed. The jars are warmed to room temperature when the extraction apparatus is ready for use. Both cartridge-filter pairs are removed from their jars, unwrapped, and placed in separate 500 ml Soxhlet extractors. Extraction is performed simultaneously using 200-300 ml of 5% diethyl ether in n-hexane (v/v) in each extractor. The Soxhlet extractors are operated for 14 to 24 hours at approximately 4 cycles per hour.

At the conclusion of the extraction process, the cleaned sampling cartridges are dried in a vacuum oven at room temperature and the two filters are discarded. The dried cartridges are wrapped in n-hexane rinsed aluminum foil and returned to their cleaned glass jar containers. The loaded jars are placed in the original shipping container and returned to the Illinois EPA Maywood office.

In the interim, the extract from the sample cartridge-filter pair is concentrated in a Kuderna-Danish evaporator. However, the trip blank extract is set aside until the sample extract is analyzed. After concentrating, the sample solution is "cleaned up" using procedures similar to those presented in Section 11 of the USEPA Method 608 (see Appendix E of this report). The final sample volume is then taken down to approximately .2 ml.

Analysis of the sample concentrate is accomplished using a gas-liquid chromatograph (GC) equipped with an electron capture detector, whose instrumental parameters are consistent with USEPA Method 608. The GC's lower detectable limit must be 500 picograms or less, and injection volumes will be between 5 and 10 microliters. If PCBs are detected in the sample, then the trip blank extract is concentrated, cleaned up, and analyzed in a manner identical to that used for the sample. If no PCBs are detected in the sample, then the trip blank extract is discarded. Sample and trip blank results will be reported by Radian in micrograms PCB as Aroclor 1242 per cartridge-filter pair.

5.0 DATA REDUCTION, VALIDATION AND REPORTING

5.1 PCB Mass Concentration

The ambient air concentration of PCBs will be calculated using the mass concentration of PCBs (see Section 4.0) and the volumetric flow rate determined in accordance with the procedures described in Section 3.0. The PCB concentrations will be expressed in micrograms per standard cubic meter (scm) of ambient air as derived from the following equation:

$$\text{PCB (as Aroclor 1242)} = \frac{\text{micrograms as Aroclor 1242}}{\text{scm of ambient air}}$$

The determination of the PCB mass in terms of Aroclor 1242 will be made according to the method described in USEPA Method 608 (see Appendix E). The sample volume will be determined after correcting the theoretical flow rate (tcfm) by the average temperature and pressure at the time of sampling and then multiplying the result by the total sample duration. The equations are as follows:

$$Qscfm = Qtcfm * (298/Tamb).5 \times (Pamb/760).5$$

$$Vscf = Qscfm * (ET)$$

Where:

Vscf = standard volume, in cubic feet.

Qscfm = the volumetric flow rate, in standard cubic feet per minute.

Tamb = ambient temperature, in degrees Kelvin.

Pamb = ambient pressure, in inches of mercury

ET = the elapsed time, in minutes.

Finally, the standard volume, Vscf, in cubic feet is converted to a standard volume, Vscm, in cubic meters, where:

$$Vscm = Vscf * .02832.$$

5.2 Data Validation

Data collected during the project will undergo several levels of review. The field sampling procedures described in Section 3 provide for the documentation of the sample collection process. Any unusual events that may affect the validity of the sample are carefully noted on these data sheets. The Radian Corporation will assist the data validation process by recording all measurements and quality control check results in bound laboratory notebooks, and by completing the chain of custody forms initiated during each sampling event. All laboratory data are routinely reviewed by Radian's quality control coordinator. The PCB detection limit of the gas chromatograph/electron capture detection procedure will be used as a basis for determining the minimum detection level for data reporting. The Project Director will certify the validity of each individual PCB sample by insuring that the appropriate sample collection and laboratory information substantiate the sample validity.

All of the data collected during the study will be compiled and reported. Anomalous data will be evaluated with the intention of resolving all possible problems. If an irreconcilable problem with the validity of a data value is encountered, the results in question will be reported and the nature of the data validity problem will be described.

5.3 Data Reporting

Pertinent data from the sample collection and laboratory analysis phases of the monitoring program will be reported on the data forms shown in Appendix F. The main product of the sampling program, the PCB mass concentration, will

be derived from the data sheets provided by the Radian Corporation and from the Illinois EPA sample collection form. The PCB concentration will be calculated as shown in Section 5.1, with the final results being entered on the appropriate data forms shown in Appendix F.

6.0 SAMPLE CUSTODY

Chain of custody procedures will be employed to the extent practical in all sample handling activities associated with this sampling program. The National Enforcement Investigation Center Policy and Procedures Manual[13] will serve as a reference in ensuring that proper sample custody protocol is maintained. The following program activities will have an inherent requirement for maintaining the chain of custody of the PCB samples.

Activity	Custodian
Preparation of Sample Cartridge	Radian Corp.
Shipment of Clean Sample Cartridge to Maywood Office	Radian Corp.
Transport of Clean Sample Cartridge to Field Site	Illinois EPA
Field Sampling	Illinois EPA

Shipment of Sample Cartridge to Austin, Texas — Illinois EPA

Laboratory Analysis — Radian Corp.

The chain of custody procedures that will be used in the sampling program are described in the following paragraphs.

6.1 General Requirements

A custodian is considered to be any person designated to provide receiving inspection and physical acceptance of a group of samples intended for subsequent treatment or analysis, analysis tracking, or sample repository operation. The custodian is charged with checking the completeness of records, maintaining sample identity, ensuring sample security and integrity, and transferring custody only under planned and controlled conditions.

6.2 Clean Cartridge Preparation and Shipment

Chain of custody procedures will originate with the preparation of the clean sample cartridge in Austin, Texas by the Radian Corp. After the cleaning operation described in Section 4 is completed, the cartridge

will be sealed in glass bottles for shipment. Each sample cartridge has a unique identifying number etched on its outer surface. The caps of each bottle will then be sealed with a strip of tamper proof, pressure sensitive, security slotted, permanent adhesive evidence tape. Radian Corp. will ship the sample cartridges in specially prepared containers by United Parcel Service.

6.3 Field Custody Procedures

6.3.1 Sample Receipt and Pre-Sampling Storage

The bottles containing the clean sample cartridges will be inspected by the field sampling team leader, Tom Barounis, upon receipt. Any damaged bottles or evidence that the tape seal has been tampered with will be grounds for not using the sample cartridge for PCB sampling. In such a case, the cartridge will not be used until it has gone through the clean-up process once more. The sample bottles and cartridges will be stored in a secure area until required for field monitoring.

6.3.2 Sampling Operations

Illinois EPA personnel will transport the sample cartridges to the field sites and install them on the sampling systems. The sample custody form shown in Figure 6.1 will be initiated at this time. Access to the sampling sites will be limited to Illinois EPA personnel and the school custodians at each site. The entry way to each roof top site will be locked and the key will remain with the building custodian. Although this will result in something of a compromise of strict chain of custody procedures, its practical impact is insignificant. After sample removal the sample custody form will be updated and the sample will be transported to Maywood.

6.3.3 Sample Packing and Shipment

Once the sample has been transported to the Maywood office, the sample will be packed for shipment to the Radian Corp. in Austin, Texas. Each sample container will be sealed with evidence tape. The chain of custody form will be completed and the original will be sent with the sample in the packing container. A copy will be maintained in the Maywood files and a copy will be sent to the Illinois EPA offices in Springfield.

CHAIN OF CUSTODY RECORD

DATE	TIME	ACTION TAKEN AND SIGNATURE

Figure 6.1 Chain-of-Custody Form

6.4 Laboratory Analysis

The Radian Corp. will acknowledge receipt of the samples by signing and dating the chain of custody form. This acknowledgement will signify that the sample has been received, that the sample numbers correspond to the chain-of-custody form and that the evidence tape has not been damaged. The samples will be registered in the laboratory log book, a sample of which is shown in Appendix F. All samples will be refrigerated in a secure area when not involved in the analytical process. The date of sample extraction and sample analysis will be entered on the chain-of-custody form at the appropriate times. This information will also be placed in the laboratory log book.

7.0 PREVENTIVE MAINTENANCE

7.1 GMW PS-1 Sampler

Preventive maintenance activities for the GMW Model PS-1 air sampler are as follows:

1. Check motor brushes before field installation of the sampler.
2. Check accuracy of the elapsed time meter before field installation and after each sampling event. Replace the meter if its time deviation is greater than ± 5 minutes.
3. Leak check the sampler prior to each sampling event.
4. Install new motors as needed.

7.2 Laboratory Equipment

The specific laboratory preventive maintenance procedures will be sufficient to meet the obligations encumbered by the Radian Corporation under its contract with the Illinois EPA. Specific terms of the work to be performed portion of the contract are shown in Appendix G. The entire contract is available upon request.

8.0 PROCEDURES FOR ASSESSING DATA ACCURACY, PRECISION, AND COMPLETENESS

The estimates of precision and accuracy of the PCB monitoring data will be made using data from similar studies as well as data collected as part of the quality assurancy activities of this monitoring program.

8.1 PCB Accuracy Estimates

8.1.1 Sampling Accuracy

The sampling accuracy of the General Metals Works PS-1 samplers will be determined from the flow rate audits conducted during the course of the study. A percent difference, D_i, between known and measured flow rates will be calculated from each audit using the equation listed in Section 3.3.1. At the end of the project the sampling accuracy, ACC, will be determined through a statistical analysis of the audit data. Sampling accuracy will be expressed as an upper and lower limit with a 95% probability of occurrence. The following relationships and equations will be used:

N = number of flowrate audits

Dav = Average of the present differences between flowrates.

S = Standard deviation of the percent differences.

$$D = \frac{\Sigma (Di)}{N} \qquad (1)$$

$$S^2 = \frac{\Sigma (Dav - Di)}{N-1} \qquad (2)$$

$$ACC = Dav + 1.96\ S \qquad (3)$$

8.1.2 Analytical Accuracy

The analytical accuracy will be determined from the spike recovery tests discussed in Section 4.2. Due to budget constraints, the spike recoveries will only be performed at the start of the project at two different Aroclor 1242 loadings. Percent differences will then be calculated from the gas chromatographic measurements versus the known loadings. A combined accuracy estimate for both loading levels will be made using equations 1 through 3 from Section 8.1.1.

8.2 PCB Precision Estimates

Estimates of sample precision will be obtained using data from a similar PCB program conducted by Battelle-Columbus in the Bloomington, Indiana area[14]. It is presumed that the precision determined in the Battelle-Columbus study will be a reasonable estimate of the precision encountered in this study.

8.3 PCB Data Completeness

The completeness of the PCB data will be determined by evaluating the number of valid samples in relation to the total number of samples planned. A sample may be rejected due to a number of reasons. Some such reasons are: broken or damaged sample containers, missing information on the data sheet, laboratory contamination, or loss in transit. The final figure for data completeness will be computed as follows:

$$\text{Completeness (\%)} = \frac{\text{Samples Received}}{\text{Samples Planned}} * 100$$

9.0 INTERNAL QUALITY CONTROL CHECKS

9.1 Sampling Equipment Checks

The sampling site operator will perform certain quality control checks for each sampling period. The elapsed time from the elapsed time meter will be compared to the start and stop time recorded on the data sheet. Any unexplained discrepancy greater than 5 minutes will result in a more detailed evaluation of each system. Prior to the installation of each sampling cartridge, the entire system will be checked for leaks.

9.2 Analytical Quality Control

9.2.1 Cartridge Cleanup Verification

A check will be performed on two of the ten cartridges that have been cleaned and prepared for initial sample collection. The selected cartridges will be re-extracted and the extract will be analyzed for PCB contamination. If the analysis shows that the PCB contamination is greater than 50 nanograms, the entire batch of cartridges will be recleaned and rechecked.

9.2.2 Blind Performance Checks

During the course of the project, two sets of exposed sampling cartridges will be spiked with known amounts of Aroclor 1242. Each of the spiked samples will be submitted to Radian as legitimate field samples, but will be flagged by the IEPA as quality control samples. The results from these checks will be used to monitor Radian's analytical performance, and to identify any apparent problems.

9.2.3 Field Blanks

Field blanks will be sent with each of the sample cartridges. The field blank will be identical to the sample cartridge in all aspects. This field blank will be carried through all field handling and laboratory operations except actual ambient sampling. If a measureable amount of PCBs is found the field sample, then the field blank will be analyzed to determine if contamination occurred during the sample collection, transport, or laboratory analysis/preparation procedure.

10.0 QUALITY ASSURANCE OBJECTIVES

The Quality Assurance objectives for the PCB sampling program are summarized in the following table.

Table 10.1 Quality Assurance Objectives for PCB Measurement Program

Detection Limit (ug/scfm)	Completeness	Precision [1]	Accuracy	Spike-Extraction Recovery	Spike Carry-over (ug)
0.0006	90%	10%	20%	90%	Less Than Detectable

1) Relative Standard Deviation

The Quality Assurance Objectives shown in Table 10.1 have been established after careful consideration of the limitations of the sampling method and procedures to be employed as well as the results of a similar study conducted in Bloomington, Indiana.[14] The degree of success obtained in meeting these quality assurance objectives will be measured using the assessment techniques described in Section 8.

11.0 PERFORMANCE AND SYSTEMS AUDIT

11.1 Systems Audit

An internal systems audit of designated project activities will be performed by the Quality Assurance Officer during the first half of the project period. The audit will consist of site inspections and a review of field monitoring practices, data recording and handling, and associated quality control activities. The procedures described in this document will be used to evaluate compliance with program objectives. Any procedural deficiencies discovered will be documented in a formal memo to the Program Manager and Project Director. The Project Director will be responsible for initiating corrective actions.

11.2 Performance Audit

Performance audits of the General Metal Works PS-1 air samplers will be performed as described in Part 3.3.1 of this monitoring plan.

12.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The Quality Assurance Officer for the project, Mr. James Buckert, will provide periodic reports of the quality assurance activities to both the Project Director, Mr. Robert Hutton, and to the Program Manager, Mr. David Kolaz. Spot inspection reports will be prepared as necessary to describe problems encountered which may affect data quality. Due to the relatively short duration of the project, the spot inspection format will be used to describe corrective actions taken for the problems that are encountered, audit results, the results of accuracy measurements, a description of the data completeness, and results of sampler calibrations.

The final report for the project will include a separate section for describing the quality control and quality assurance procedures, the results of the internal quality control checks, the precision and accuracy assessments, and an overall evaluation of the study results in terms of the quality assurance objectives.

13.0 CORRECTIVE ACTION STRATEGY

Problems that may affect the quality of the data acquired during the term of the sampling program will be immediately corrected. Any such problems requiring corrective action will be brought to the attention of the Program Manager, the Project Director, and the Quality Assurance Officer. The individual identifying the problem should report it to the above individuals, in writing, with the recommended corrective action. Due to the complexity and diversity of the sampling and analysis system it would be impracticable to attempt to predict all the potential problems and corrective actions which may be required. The following table identifies some of the more likely problems and their corrective actions.

Table 13.1 Corrective Action Summary

System	Problem/QC Limit	Corrective Action
GMW PS-1 Sampler	System Leak	Check Gaskets/Repair Leak
	Timer Malfunction/ Failure	Repair/Replace Timer
	Elapsed Time Meter Malfunction/Failure	Repair/Replace
	Motor Failure	Repair/Replace
	Sampler Flow Rate More than 10% from Audit Flow Rate	Recalibrate Sampler
	Magnehelic Gauge Malfunction/Failure	Recalibrate/Replace

System	Problem/QC Limit	Corrective Action
	Major damage or failure of any structural or operational component of sampler	Repair/Replace
PCB Analysis	Cleaned Glass Cartridge Damaged	Replace
	Sample Cartridge Received in Damaged Condition or Damaged Upon Receipt	Analyze if Possible, Remove Cartridge from Program

The above corrective actions and any others required in this program necessitate an assessment of the impact of the problem/failure on data validity. Therefore, inherent in any corrective action will be a determination by the Project Director, with input from the Quality Assurance Officer, of the affect on the data. The data will then be considered as valid, questionable, or invalid.

Any corrective action required by Table 13.1 or any other activity required to overcome a problem or situation that may jeopardize the quality of the data acquired during the program is the responsibility of the person reporting and recognizing the situation. The Project Director, Mr. Robert Hutton, will be responsible for approving the appropriate corrective action for the Illinois EPA and for follow-up to ensure that the action resolves the problem. Maureen McDevitt will be responsible for any corrective action that may be required by the Radian Corporation in the laboratory analysis of the PCB samples.

14.0 RESOURCE REQUIREMENTS

14.1 Equipment Requirements

The following items of equipment will be dedicated to the PCB sampling project.

GMW PS-1 pesticide and vapor collection system (2): $3,000

GMW Model 40 calibration orifice: $400

14.2 Contractual Requirements

The analytical portion of the PCB analysis will be performed under contract to the Radian Corporation. The work to be performed portion of the contract is presented in Appendix F. The total amount of the contract is $10,000.

14.3 Commodity Requirements

The following items will be maintained to support the field sampling portion of the PCB sampling program. All necessary supplies required for the laboratory portion of the project are considered an integral part of the contractual arrangements with the Radian Corp.

Ten sampling cartridges: $650

Polyurethane foam plugs: $80

Florosil: $150

Spare motor: $80

Spare timer: $75

Spare elapsed time meter: $60

Spare Magnehelic gauge: $40

Spare shelter: $600

Shipping containers: $150

Rubber gloves: $60

14.4 Manpower Requirements

The collection of 16 PCB samples will require approximately 4 man-weeks including the packing and shipping of the samples to the Radian Corp. in Austin, Texas. The installation and preparation of the sampling sites at Bright Elementary School and Grissom Elementary School will require an additional 2 man-weeks. Contract management, negotiations, preparation, and project management will require approximately 4 man-weeks. Preparation of the final report will require 6 man-weeks. The total manpower requirements for this project, excluding the laboratory portion, will be 4 man-months.

15.0 REFERENCES

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4. 1980 Census of Population and Housing, U.S. Department of Commerce, Issued November 1981.

5. "The Southeast Chicago Study: An Assessment of Environmental Pollution and Public Health Impacts", Illinois Environmental Protection Agency (Draft April 1984).

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8. Lewis, R.G. and Jackson, M.D., "Modification and Evaluation of a High Volume Air Sampler for Pesticides in Semivolatile Industrial Oranic Chemicals", Analytical Chemistry, 54, No. 3, pp. 592-594 (March, 1982).

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10. You, F., Bidleman, T.F., "Influence of Volatility on the Collection of Polycyclic Aromatic Hydrocarbon Vapors with Polyurethane Foam", Environmental Science and Technology, 18, No. 5, pp. 330-333 (May 1984).

11. Federal Register, Volume 44, No. 233, Monday, December 3, 1979, pp. 69501-69509.

12. Smith, F., Wohlschlegel, P.S., Rogers, R.S.C., and Mulligan, D.J., "Investigation of Flow Rate Calibration Procedures Associated with the High Volume Method for Determination of Suspended Particulates", Research Triangle Institute and North Carolina State University, EPA Report No. 600/4-78-047 (August, 1978).

13. National Enforcement Investigation Center (NEIC) Policy and Procedures Manual, EPA-330/9-78-001-R, May, 1978 (Revised October, 1979).

14. Battelle-Columbus Laboratories, "Quality Assurance Plan for Ambient Monitoring for PCBs Near Three Landfills in the Bloomington, Indiana Area", Final Report to the USEPA, Contract No. 68-02-3745, Work Assignment No. 4 (March 31, 1983).

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APPENDIX A

SCA Chemical Waste Incinerator
PCB Stack Test Results

SCA Chemical Waste Incinerator
PCB Stack Test Results

	PCB Feed (lb/Min)	PCB Emissions (µg/sec)	PCB Destruction and Removal Efficiency (%)
Run 10*	12	19	99.99998
Run 11	21	16	99.99999
Run 12	7	26	99.99995
Run 13	19	14	99.99999
Run 14	98	297	99.99996
Run 17**	33	450	99.99982
Run 19	47	233	99.99994
Run 20	34	1267	99.99949
Run 21	29	433	99.99980

* Runs 10-14 were conducted in October 1982.
** Runs 17-21 were conducted in July 1984.

APPENDIX B

Predicted Worst Case PCB Concentrations at the Orville T. Bright and Virgil S. Grissom Elementary Schools Using the Real Time Air Quality Model (RAM)

The worst case PCB concentration expected to occur at the Bright and the Grissom Elementary Schools sampling sites was computed using a mathematical dispersion model. This model, known as the Real Time Air Quality Model (RAM), computed the 24 hour concentration at the two schools using hourly meteorological conditions obtained from O'Hare Airport for calendar year 1975. Specific information on the dispersion model can be obtained from Turner, D.B., and Novak, J.H., "Users Guide for RAM", Environmental Protection Agency, Research Triangle Park, NC (1978).

The source information input to the model is contained in the following table.

Source Parameter Input to RAM

STACK HEIGHT (Meters)	STACK DIAMETER (Meters)	STACK TEMPERATURE (Kelvin)	STACK GAS EXIT VELOCITY (Meters/Sec)	PCB EMISSION RATE (Micrograms/Sec)
25.3	2.1	338	6.5	297

The PCB emission rate from the preceding table represents the highest emission rate found from the stack tests of the incinerator which still showed a destruction and removal efficiency of 99.9999%.

The Bright Elementary School is located 2.3 kilometers from the SCA incinerator on a 33 degree (northeast) line. Grissom Elementary School is located on a line 139 degrees (southeast) from SCA at a distance of 3.0

kilometers. The Bright Elementary School sampling height is 17.8 meters above ground level while the Grissom Elementary School sampler is 7.6 meters above grade. Both sampling heights were included in the dispersion model calculations.

The results of the dispersion calculations are shown in the following table.

Calculated PCB Concentrations*
(Micrograms per Cubic Meter)

Site	Highest	5th Highest
Bright	.0008	.00029
Grissom	.00016	.00010

*PCB Emission Rate = 297 µg/sec.

The 5th highest concentration shown in the previous table gives an indication of how fast the ambient concentrations fall off. It is apparent that the Bright Elementary School site has the potential of measuring the highest concentration. The minimum detectable limit of the sampling methodology is 0.0006 micrograms per cubic meter (see Section 10). Therefore, if the worst case meteorological condition occurs while SCA is emitting PCBs at the maximum rate shown, detectable levels of PCBs would be measured at Bright Elementary School. The typical case PCB emission rate is 74 µg/sec which would result in no detectable PCB concentrations at either site.

If the absolute highest emission rate of PCBs is taken from the stack test results shown in Appendix A, the following results are obtained.

Calculated PCB Concentrations with
a PCB Emission Rate of 1267 µg/sec
(Micrograms per Cubic Meter)

Site	Highest	5th Highest
Bright	.0034	.0012
Grissom	.0007	.0004

The results of the preceding table show that the highest emission rate measured from the SCA stack tests (Appendix A) would produce measurable PCB concentrations at both the Bright and Grissom Elementary School sites.

The conclusion that can be drawn from this analysis is that detectable concentrations of PCBs at the two Elementary school sampling sites are unlikely to occur with the SCA incinerator operating under permitted conditions. Operating at the maximum allowed tested rate of 297 µg/sec, the predicted increase in cancer would be 1 in 11,800,000 in the area of the Bright Elementary School.[6]

APPENDIX C

Orifice Calibration Unit Certification Data

CALIBRATOR

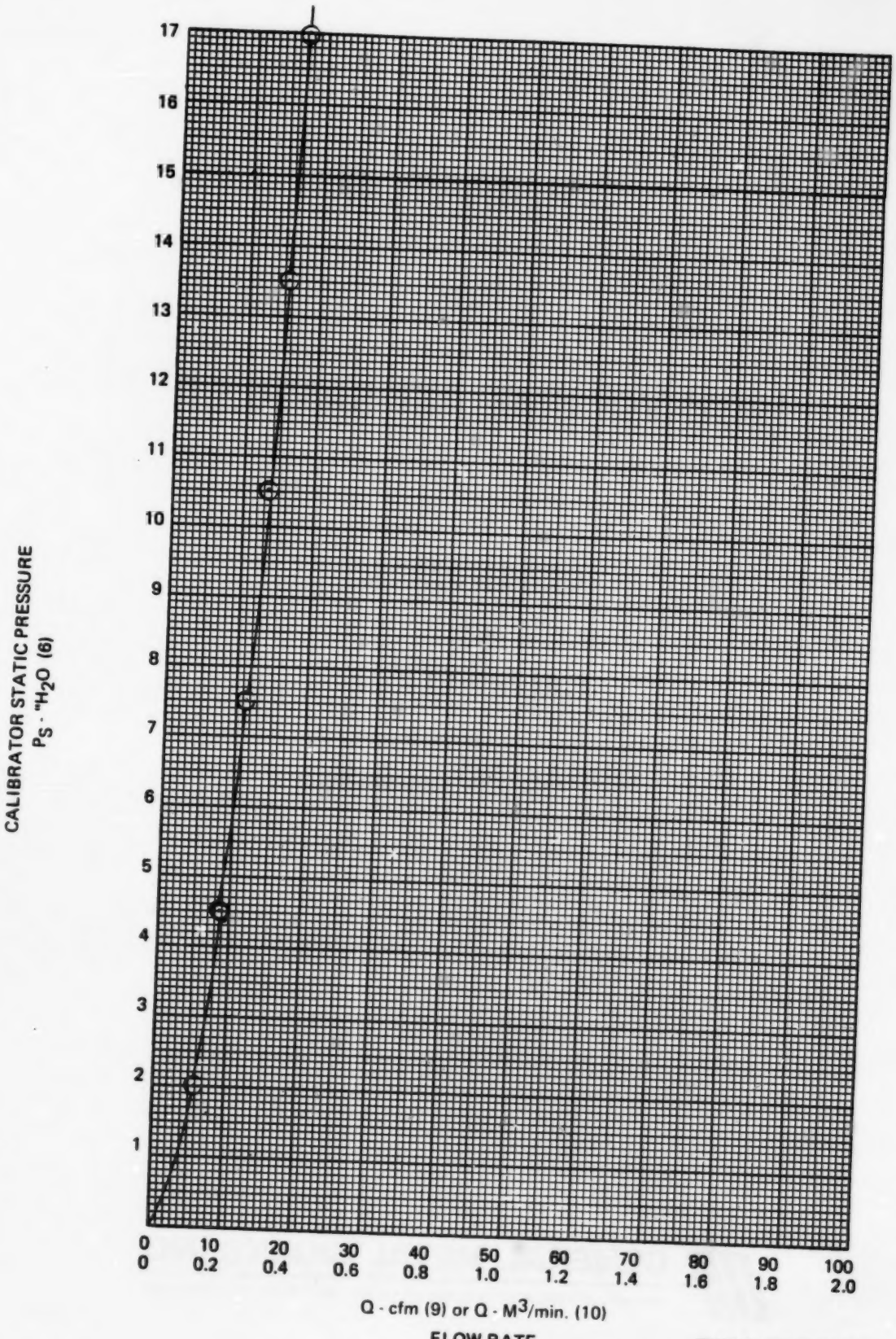
for

HIGH VOLUME AIR SAMPLER

CERTIFICATE
of
CALIBRATION

SERIAL NO. 30 B

GENERAL METAL WORKS INC.
8368 BRIDGETOWN ROAD / VILLAGE OF CLEVES, OHIO 45002 / TEL. 513-941-2229



THIS PLOT IS IN (check one)
cfm ✓
M^3/min. _____
They are NOT EQUIVALENT

GMW Model 40 Orifice Calibration Table

Orifice Unit Number: 30B
Calibration Date: 03-06-84
Calibration Agency: General Metals Works, Inc.
Verified by: James Buckert

Data Table

Manometer (in. H_2O)	Flowrate (tcfm)	Manometer (in. H_2O)	Flowrate (tcfm)	Manometer (in. H_2O)	Flowrate (tcfm)
10.9	13.65	7.9	11.60	4.9	9.11
10.8	13.58	7.8	11.53	4.8	9.02
10.7	13.52	7.7	11.45	4.7	8.92
10.6	13.46	7.6	11.38	4.6	8.83
10.5	13.39	7.5	11.30	4.5	8.73
10.4	13.33	7.4	11.23	4.4	8.63
10.3	13.26	7.3	11.15	4.3	8.53
10.2	13.20	7.2	11.07	4.2	8.43
10.1	13.13	7.1	10.99	4.1	8.33
10.0	13.07	7.0	10.91	4.0	8.22
9.9	13.00	6.9	10.84	3.9	8.12
9.8	12.93	6.8	10.76	3.8	8.01
9.7	12.87	6.7	10.68	3.7	7.90
9.6	12.80	6.6	10.59	3.6	7.79
9.5	12.73	6.5	10.51	3.5	7.68
9.4	12.67	6.4	10.43	3.4	7.57
9.3	12.60	6.3	10.35	3.3	7.46
9.2	12.53	6.2	10.27	3.2	7.34
9.1	12.46	6.1	10.18	3.1	7.23
9.0	12.39	6.0	10.10	3.0	7.11
8.9	12.32	5.9	10.01	2.9	6.98
8.8	12.25	5.8	9.92	2.8	6.86
8.7	12.18	5.7	9.84	2.7	6.74
8.6	12.11	5.6	9.75	2.6	6.61
8.5	12.04	5.5	9.66	2.5	6.48
8.4	11.97	5.4	9.57	2.4	6.34
8.3	11.89	5.3	9.48	2.3	6.21
8.2	11.82	5.2	9.39	2.2	6.07
8.1	11.75	5.1	9.30	2.1	5.93
8.0	11.68	5.0	9.21	2.0	5.78

$Q(theo) = K \times (H)^{.5} + b$

$K = 4.168 \qquad b = -0.114$

Correlation Coefficient = .9999

JB:jd/1771D/1,sp

(9) and (10) are corrected to 760 mm of Hg and 25°C

CALIBRATION WORK SHEET

(1) Test-Point No.	(2) Elapsed Time - Δt Min.	(3) Initial Volume V_{in} M^3	(4) Meter Static Pressure - P_S "Hg	(5) True Volume V_T M^3	(6) Calibrator Static Press. "H_2O	(7) Flow Rate Q M^3/min.	(8) Flow Rate Q ft^3/min.	(9) Corrected Flow Rate Q ft^3/min.	(10) Corrected Flow Rate Q M^3/min.
1	5.998	1	0.2	0.993	2.0	0.166	5.8	5.8	0.165
2	3.989	1	0.4	0.986	4.5	0.247	8.7	8.7	0.247
3	3.068	1	0.6	0.980	7.5	0.319	11.3	11.2	0.318
4	2.562	1	0.8	0.973	10.5	0.380	13.4	13.4	0.379
5	2.241	1	1.0	0.966	13.5	0.431	15.2	15.2	0.430
6	1.972	1	1.2	0.959	17.0	0.486	17.2	17.1	0.485
7									

(11) P_B: 29.44 "Hg

(12) T: 71° °F

(13) RH: 42 %

Roots Meter No.: 7509364

Calibrator:
Model No.: GMW- Spec.

Serial No.: 30 B

Calibration performed by: Brian Barry

Date of Calibration: 3/6/84

Date placed in service: _____
(To be noted by user)

EQUATIONS

$$V_T = V_{in} \frac{P_B - P_S}{P_B}$$

$$= (3) \frac{(11) - (4)}{(11)}$$

$$Q = \frac{V_T}{\Delta t} = \frac{(5)}{(2)}$$

$$M^3 \times 35.31 = Ft^3$$

When using this instrument to calibrate a high volume sampler at conditions other than those at which the calibration took place, or was corrected to, (11) (12), the following relation should be applied.

$$Q_T = Q \left[\frac{T_T}{T} \frac{P_B}{P_T} \right]^{1/2}$$

NOTE: IN THIS EQUATION USE ABSOLUTE TEMPERATURE (RANKINE)

where:

Q_T = flow at new conditions T_T and P_T.

For additional information consult:

1. The Federal Register, Vol. 36, No. 84, pp. 8191 - 8194, April 30, 1971.
2. Guidelines for Development of a Quality Assurance Program – High Volume Method. EPA - 600/4-77-027a

Notes:
1. Calibrators should be recalibrated after one year of field use.
2. Copies of this calibration are not kept on file.

APPENDIX D

Standard Operating Procedures for the Calibration and Operation of the GMW Model PS-1 Air Sampler

TITLE: Generation of the PS-1 Calibration Table

1. Purpose:

   To review the PS-1 field calibration data and generate a printed data table relating measured flowrates to observed magnehelic gauge readings.

2. Applicability:

   This procedure is applicable to the creation of PS-1 calibration tables using the IBM PC computer.

3. Responsibilities:

   Personnel who review calibration data will be knowledgeable of this SOP.

4. References:

   4.1 IBM Disk Operating System, Version 2.00, Microsoft, Inc., 1983.
   4.2 IBM Basic, Version 1.10, Microsoft, Inc., 1982.
   4.3 Ambient Air Monitoring Section SOP QA-050.

5. Equipment:

   5.1 IBM PC.
   5.2 IBM PC graphics printer.

6. Procedure:

   NOTE: A working knowledge of the IBM PC and DOS systems is required for this SOP.

   6.1 Preliminary Data Review:

   1. Upon receipt of the GMW Model PS-1 Calibration Form, review all data entries for completeness. If a critical parameter is missing, then void the submittal and request a recalibration.
   2. Review the entries against the quality control limits listed in SOP FO-050, part 6.2. If any limit is exceeded, then the calibration is void and a recalibration must be performed.

   6.2 Generation of Calibration Table:

   1. Activate the IBM printer and boot the IBM PC under the DOS operating system.
   2. Enter the Advanced Basic mode by keying in -- basica /d -- when the appropriate DOS prompt is present. /d is used to provide double precision for transcendental functions.

3. Obtain a diskette containing the PS1CAL.BAS program and insert it into drive B. Load and run the program by keying in -- load "b:pslcal",r.
4. As soon as the program begins, the CRT screen is cleared and the following question displayed: PS-1 Shelter No.?
5. Immediately key in the sampler's IEPA tag number. Do not key in more than 6 digits. Press the "enter" key to enter the number.
6. The CRT now displays the following question: Calibration Data (mm/dd/yy)?
7. Immediately key in the month, day, and year of the calibration. Fox example, if the calibration was performed on January 5, 1984, then key in 01/05/84. Do not key in more than 8 symbols. Press the "enter" key to enter the number.
8. The CRT returns with the question: Expiration Date (mm/dd/yy)?
9. Key in the month, day, and year after which the calibration results will be considered void. A calibration is only valid for six months. If the date of calibration is January 5, 1984, then the expiration date is July 5, 1984. This should be keyed in as 07/05/84. Press the "enter" key to enter the number.
10. The question, "Number of Calibration Points?", is now displayed on the screen. Count the number of magnehelic display entries on the calibration form. Disregard the first display entry at 70. If a display entry at 0 is not listed, then add it to your count. Immediately key in your count number. Press the "enter" key to enter the number.
11. The CRT now reads: Magnehelic Value, Flowrate (tcfm)?
12. Review the PS-1 calibration data but disregard the first magnehelic gauge/flowrate data pair (i.e., magnehelic gauge at 70). Key in one of the magnehelic gauge values, followed by a comma, then the corresponding flowrate. For example, a magnehelic gauge value at 60, with a flowrate of 7.75 tcfm, would be keyed in as -- 60,7.75. Press the "enter" key to enter the data pair.
13. Repeat step 6.2.12 for the remaining data pairs. If the magnehelic value at 0, flowrate of 0, is not on the calibration form, key them in anyway.
14. When the final data pair is entered, the screen will display the linear regression results, calibration data, and expiration date.
15. If the correlation coefficent is greater than or equal to 0.9990, then a calibration table is printed. If the coefficient is less than 0.9990, then the calibration is unacceptable and the table is not printed. The statement, "The correlation coefficient is less than 0.9990", is printed instead of the table.

NOTE: If the calibration is unacceptable, then another one must be performed as soon as possible.

TITLE: Generation of the PS-1 Calibration Table

16. After the calibration table is printed, the screen will display the question: Do you want another table printed? -- and -- Enter Y or N. Key in a Y for Yes or an N for no, and press the "enter" key. If Y is entered, then the same question will reappear after the table is generated. You can generate as many replicate tables as required by entering a Y each time the question appears. Entering an N will terminate the table generation routine.
17. When all replicate tables have been made and the N entered in step 6.2.16, the CRT will display: Are there additional PS-1 calibrations to be calculated? -- and -- Enter Y or N. This question is also displayed after step 6.2.15 if the correlation coefficient was less than 0.9990. In response, key in a Y for yes or an N for no, and press the "enter" key. If Y is entered, then the program will restart itself beginning at step 6.2.4. If N is entered, the program will terminate.
18. After all calibration tables have been generated, remove the program diskette from drive B and return it to its protective envelope. Also remove the data tables from the printer, and turn off the IBM PC and printer.
19. On each calibration table identify the sampling site by writing in the appropriate city, location, and address in the spaces provided. Also sign your name and indicate the current date in the spaces marked Originator and Date.
20. Return one copy of the completed calibration table to the field operator. Retain a replicate copy with the PS-1 calibration form.

JB:bv/sp/1548D/1-3

PS-1 Calibration Table

Sampler No. - 13162
Calibration Date - 07/25/84
Slope - 1.2159
Intercept - 0.0535
Std. Error - 0.1193
Corr. Coeff. - 0.9996

City _____

Location _____

Address _____

Magnehelic Reading	Flowrate (tcfm)	Magnehelic Reading	Flowrate (tcfm)	Magnehelic Reading	Flowrate (tcfm)
70	10.23	56	9.15	42	7.93
69	10.15	55	9.07	41	7.84
68	10.08	54	8.99	40	7.74
67	10.01	53	8.91	39	7.65
66	9.93	52	8.82	38	7.55
65	9.86	51	8.74	37	7.45
64	9.78	50	8.65	36	7.35
63	9.70	49	8.56	35	7.25
62	9.63	48	8.48	34	7.14
61	9.55	47	8.39	33	7.04
60	9.47	46	8.30	32	6.93
59	9.39	45	8.21	31	6.82
58	9.31	44	8.12	30	6.71
57	9.23	43	8.03	29	6.60

This calibration table is void after 01/25/85

Originator _____ Date _____

```
1000 REM      ************************************************************************
1010 REM      *   PROGRAM NAME -- PS1CAL.BAS  (Ver. 1.0) - AUGUST,1984              *
1020 REM      *   AUTHOR -- Jim Buckert              IEPA-APC-AAM                    *
1030 REM      *                                                                      *
1040 REM      *   This program is written in IBM Advanced BASIC. It is a             *
1050 REM      *   modification of the program HVOLCAL.BAS (Ver. 1.0) written         *
1060 REM      *   by Dennis Bomke.  The program uses field calibration data          *
1070 REM      *   for PS-1 samplers.  Summary statistics are calculated and          *
1080 REM      *   checked against established limits.  Data tables can be            *
1090 REM      *   generated if the quality control limits are met.                   *
1100 REM      ************************************************************************
1110 REM
1120 REM ***************************************************************************
1130 REM **                  DEFINE AND INITIALIZE VARIABLES                      **
1140 REM ***************************************************************************
1150 DEFINT N
1160 DEFDBL C-E,S,X,Y
1170 SUMX = 0!
1180 SUMY = 0!
1190 SUMSQX = 0!
1200 SUMSQY = 0!
1210 SUMXY = 0!
1220 CORCOF = .999
1230 CLS
1240 REM ***************************************************************************
1250 REM **                          INPUT CALIBRATION DATA                       **
1260 REM ***************************************************************************
1270 INPUT "PS-1 Shelter No.     ";HVMOTN$
1280 INPUT "Calibration Date (mm/dd/yy)      ";CDATE$
1290 INPUT "Expiration Date (mm/dd/yy)       ";EXPIR$
1300 INPUT "Number of Calibration Points     ";N
1310 PRINT
1320 FOR I=1 TO N
1330 INPUT "Magnehelic Value, Flowrate(tcfm) "; X(I), Y(I)
1340 NEXT
1350 REM ***************************************************************************
1360 REM **       ACCUMULATE NECESSARY SUMS TO BE USED IN CALCULATION            **
1370 REM **                    THE EQUATION IS OF THE FORM:                      **
1380 REM **    FLOWRATE = SLOPE X (SQUARE ROOT(MAGNEHELIC)) + INTERCEPT          **
1390 REM ***************************************************************************
1400 FOR I=1 TO N
1410 SUMX=SUMX + SQR(X(I))
1420 SUMY=SUMY + Y(I)
1430 SUMSQX=SUMSQX + X(I)
1440 SUMSQY=SUMSQY + (Y(I))^2
1450 SUMXY=SUMXY + Y(I) * SQR(X(I))
1460 NEXT
1470 REM ***************************************************************************
1480 REM **                      CALCULATE SUMMARY STATISTICS                     **
1490 REM ***************************************************************************
1500 DENOM = N*SUMSQX - SUMX^2
1510 SLOPE = (N*SUMXY - SUMX*SUMY)/DENOM
1520 ENTRCP = (SUMSQX*SUMY - SUMX*SUMXY)/DENOM
1530 STNDER = SQR((SUMSQY - ENTRCP*SUMY - SLOPE*SUMXY)/(N - 2))
1540 CORR = (N*SUMXY - SUMX*SUMY)/SQR(DENOM*(N*SUMSQY - SUMY^2))
1550 REM ***************************************************************************
1560 REM **              PRINT RESULTS TO THE SCREEN FOR REVIEW                   **
1570 REM ***************************************************************************
1580 PRINT:PRINT
```

```
1590 PRINT USING "SLOPE =       #.####    ";SLOPE
1600 PRINT USING "INTERCEPT =   #.####    ";ENTRCP
1610 PRINT USING "STANDARD ERROR =              #.####    ";STNDER
1620 PRINT USING "CORRELATION COEFFICIENT =   #.####    ";CORR
1630 PRINT "CALIBRATION DATE WAS:      ";CDATE$
1640 PRINT "EXPIRATION DATE WAS:       ";EXPIR$
1650 REM ************************************************************************
1660 REM **                          PRINT TABLE HEADINGS                      **
1670 REM ************************************************************************
1680 LPRINT CHR$(12):LPRINT:LPRINT:LPRINT:LPRINT
1690 LPRINT CHR$(14); CHR$(27); CHR$(69); "          PS-1 Calibration Table"
1700 LPRINT:LPRINT:LPRINT:LPRINT CHR$(10); CHR$(27); CHR$(70)
1710 LPRINT USING "    Sampler No. -              \     \ ";HVMOTN$
1720 LPRINT USING "    Calibration Date -     \       \ "; CDATE$;
1730 LPRINT "   City _____________________________________"
1740 LPRINT USING "      Slope -                  #.#### "; SLOPE
1750 LPRINT USING "      Intercept -              #.#### "; ENTRCP;
1760 LPRINT "   Location _________________________________"
1770 LPRINT USING "      Std. Error -             #.#### "; STNDER
1780 LPRINT USING "      Corr. Coeff. -           #.#### "; CORR;
1790 LPRINT "   Address __________________________________"
1800 LPRINT:LPRINT:LPRINT:LPRINT
1810 REM ************************************************************************
1820 REM **                        DETERMINE ACCEPTABILITY                     **
1830 REM ************************************************************************
1840 IF CORR<CORCOF THEN GOTO 2250
1850 REM ************************************************************************
1860 REM **                     CALCULATE AND PRINT DATA TABLE                 **
  70 REM ************************************************************************
1880 LPRINT "    Magnehelic  Flowrate      Magnehelic  Flowrate      Magnehelic
 Flowrate"
1890 LPRINT "      Reading     (tcfm)         Reading     (tcfm)         Reading
  (tcfm)"
1900 LPRINT "    ---------------------     ---------------------     ----------
---------"
1910 FOR I=1 TO 14
1920 MAG1 = 71 - I
1930 MAG2 = 57 - I
1940 MAG3 = 43 - I
1950 FLR1 = SLOPE * SQR(MAG1) + ENTRCP
1960 FLR2 = SLOPE * SQR(MAG2) + ENTRCP
1970 FLR3 = SLOPE * SQR(MAG3) + ENTRCP
1980 LPRINT USING "         ##";MAG1;
1990 LPRINT USING "       ##.##  "; FLR1;
2000 LPRINT USING "          ##";MAG2;
2010 LPRINT USING "       ##.##  "; FLR2;
2020 LPRINT USING "          ##";MAG3;
2030 LPRINT USING "       ##.##"; FLR3
2040 NEXT I
2050 LPRINT "    ---------------------     ---------------------     ----------
---------"
2060 LPRINT:LPRINT:LPRINT:LPRINT CHR$(27);CHR$(69);
2070 LPRINT USING "                    This calibration table is void after \
  ;EXPIR$
2080 LPRINT CHR$(27);CHR$(70);
2090 LPRINT:LPRINT:LPRINT:LPRINT "    Originator ____________________     Date _
--------------"
```

```
2100 REM ************************************************************************
2110 REM **                          PRINT ADDITIONAL TABLES                   **
 '20 REM ************************************************************************
 .30 PRINT:PRINT "Do you want another table printed ?"
2140 INPUT "Enter Y or N ";ANS$
2150 IF ANS$ = "Y" OR ANS$ = "y" GOTO 1660
2160 REM ************************************************************************
2170 REM **                  CHECK FOR ADDITIONAL CALIBRATIONS                 **
2180 REM ************************************************************************
2190 PRINT:PRINT  "Are there additional PS-1 calibrations to be calculated ?"
2200 INPUT "Enter Y or N       ";ANS$
2210 IF ANS$ = "Y" OR ANS$ = "y"    GOTO 1170    ELSE END
2220 REM ************************************************************************
2230 REM **        ROUTINES TO PRINT OUT OF TOLERANCE DESCRIPTION              **
2240 REM ************************************************************************
2250 LPRINT:LPRINT:LPRINT
2260 LPRINT CHR$(27);CHR$(45);CHR$(1);  SPC(10) "The correlation coefficient is
less than 0.9990"
2270 LPRINT CHR$(27);CHR$(45);CHR$(0)
2280 GOTO 2190
```

AMBIENT AIR MONITORING STANDARD OPERATING PROCEDURES

NUMBER: FO-050
DATE: Jan. 2, 1985
PAGE: 1 of 7
Revision No.: 2

TITLE: Calibration of the GMW Model PS-1 Air Sampler

1. Purpose:

   To establish the response of the magnehelic gauge/venturi system used in the GMW Model PS-1 air sampler against known flowrates as measured by a GMW Model 40 orifice calibration unit.

2. Applicability:

   This procedure is applicable to the field calibration of Model PS-1 samplers over the flowrate range of 6.00 to 17.00 theoretical cubic feet per minute (tcfm). Each sampler is calibrated initially and ever six months thereafter, upon replacement of a venturi or magnehelic gauge, or when a one point flowrate audit near 8 tcfm exceeds ±10% difference.

3. Responsibilities:

   Personnel performing or evaluating field calibrations will be knowledgeable of this SOP.

4. References:

   4.1 Operating Instructions, Model PS-1 (published by General Metal Works, Inc.).
   4.2 Investigation of Flow Rate Calibration Procedures Associated with the High Volume Method for Determination of Suspended Particulates, EPA-600/4-78-047, August 1978.
   4.3 40 CFR, Part 50, Appendix B.

5. Equipment:

   5.1 GMW Model 40 orifice calibration unit with water manometer, manometer accurate to within ± 0.05 inch.
   5.2 GMW Model PS-1 sampler.
   5.3 Thermometer, accurate to within ± 0.5°C.
   5.4 Barometer, accurate to within ± T mmHg.

6. Procedure:

   NOTE: Record calibration data on the GMW Model PS-1 Calibration Form, see Attachment A.

TITLE: Calibration of the GMW Model PS-1 Air Sampler

6.1 Pre-calibration:

1. Phone the DMA Unit in Springfield and obtain the station pressure, in mmHg, at the sampler's location.
2. Open the PS-1 sampler hood and secure it to the back latch.
3. Attach the thermometer and manometer support braces to the top edge of the shelter.
4. Attach the thermometer and manometer to their respective support braces.
5. Open both ports on the manometer by turning the L-connectors 3/4 revolution counter-clockwise, then connect a 2' section of 3/16" I.D. latex hose to one of the ports.
6. Check the manometer liquid for free movement against pressure and adjust the manometer scale to zero.

NOTE: Refer to figures 1 and 2 for identification of the sampler components.

7. Remove the polyethylene cover from the aluminum sampling module, then disconnect the module from the sampler's pneumatic line.
8. Check the meter zero on the sampler's magnehelic gauge and adjust to zero if necessary.
9. While holding the sampling module in an up-right position, unscrew and remove the lower canister from the filter holder support.
10. Hand tighten the module's filter holder support/filter holder connection.
11. Check for the presence of a gasket in the bottom of the lower canister, and also in the base of the filter holder support. If either gasket is missing, install another before proceeding.
12. Place an empty glass cartridge (2.5" O.D. x 5.25" length) in the lower canister, then reconnect the canister to the filter holder support.

Caution: Do not attempt to over-tighten the canister/filter holder support connection. Hand tighten only. Too much force will break the glass cartridge.

13. Reconnect the sampling module to the sampler's pneumatic line by applying torque only to the module's lower canister. Hand tighten only.
14. Remove the filter retaining ring from the filter holder.
15. Place the GMW Model 40 orifice calibration unit (OCU) on the filter holder and secure it to the holder by tightening the three swing bolts.

16. Fully open the sampler's ball-valve.
17. Engage the sampler's power switch, located on the Paragon timer.
18. With a screwdriver, adjust the sampler's voltage control screw (located next to the elapsed time meter) to obtain a magnehelic gauge reading of 100.
19. Plug the OCU's top opening with a No. 0 rubber stopper, and the OCU's side arm port with a finger. The sampler's magnehelic gauge should read exactly zero.

NOTE: If the magnehelic reading is above zero, then an air leak is present. Eliminate any leak before continuing. If the magnehelic display is below zero, then contact the Springfield headquarters before proceeding. Record all actions on the calibration form.

20. Upon completion of the leak check, unplug the OCU, then disengage the sampler's power switch.
21. Connect the free end of the 3/16" I.D. latex hose on the manometer to the OCU side arm port.
22. Re-engage the sampler's power switch and allow the system to warm up for 10 minutes.

6.2 Calibration:

1. Slightly close the sampler's ball valve until the magnehelic gauge is at 70. Record the magnehelic display, water manometer displacement to within $\pm$ 0.05 inch, and thermometer reading to within + 1°C.
2. Repeat step 6.2.1 for magnehelic gauge readings at 60, 50, 40, 30, and 70 units, respectively.

Q.C. Check: If any of the following quality control limits are exceeded, then the calibration is void:

(a) The ambient temperature must be at least 5°C, but not greater than 38°C,
(b) The difference between the maximum and minimum temperature measured during the calibration cannot exceed 5°C, and
(c) The difference between the initial and final water manometer displacements (magnehelic gauge readings at 70) cannot exceed $\pm$ 0.15 inch.

6.3 Post-calibration:

1. Disengage the sampler's power switch.
2. Disconnect the latex hose from the manometer and OCU, then close the manometer ports.
3. Remove the OCU manometer, thermometer, and manometer and thermometer support braces.
4. Reattach the filter retaining ring to the filter holder.
5. Disconnect the aluminum sampling module from the sampler's pneumatic line.
6. While holding the sampling module in an up-right position, unscrew and remove the lower canister from the filter holder support.
7. Remove the empty glass cartridge from the lower canister, then reconnect the canister to the filter holder support.
8. Reconnect the sampling module to the sampler's pneumatic line.
9. Cover the sampling module with a clean polyethylene bag.
10. Close and secure the shelter's hood.
11. Complete the calibration form and return it to the DMA Unit in Springfield.

JB:jd/1435D/1,4/sp

GMW MODEL PS-1 CALIBRATION FORM

Name: _____ Date: _____

Site Address: _____

PS-1 Shelter No.: _____ Station Pressure: _____

GMW Model 40 OCU No.: _____

Magnehelic Gauge Reading	Manometer Reading (in. H_2O)	OCU Flow-Rate (tcfm)	Temp. (^oC)

Comments: _____

_____

_____

_____

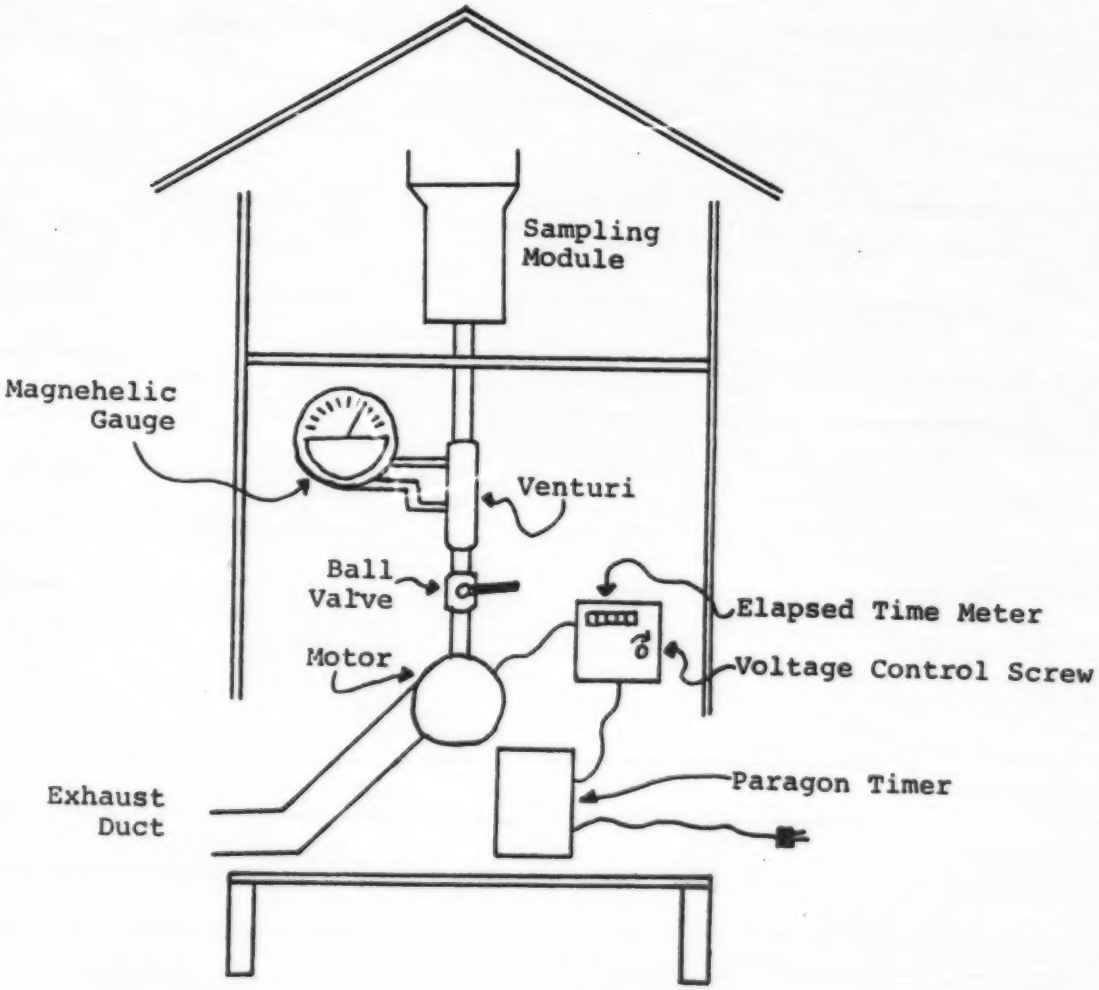


Figure 1 GMW Model PS-1 Sampler

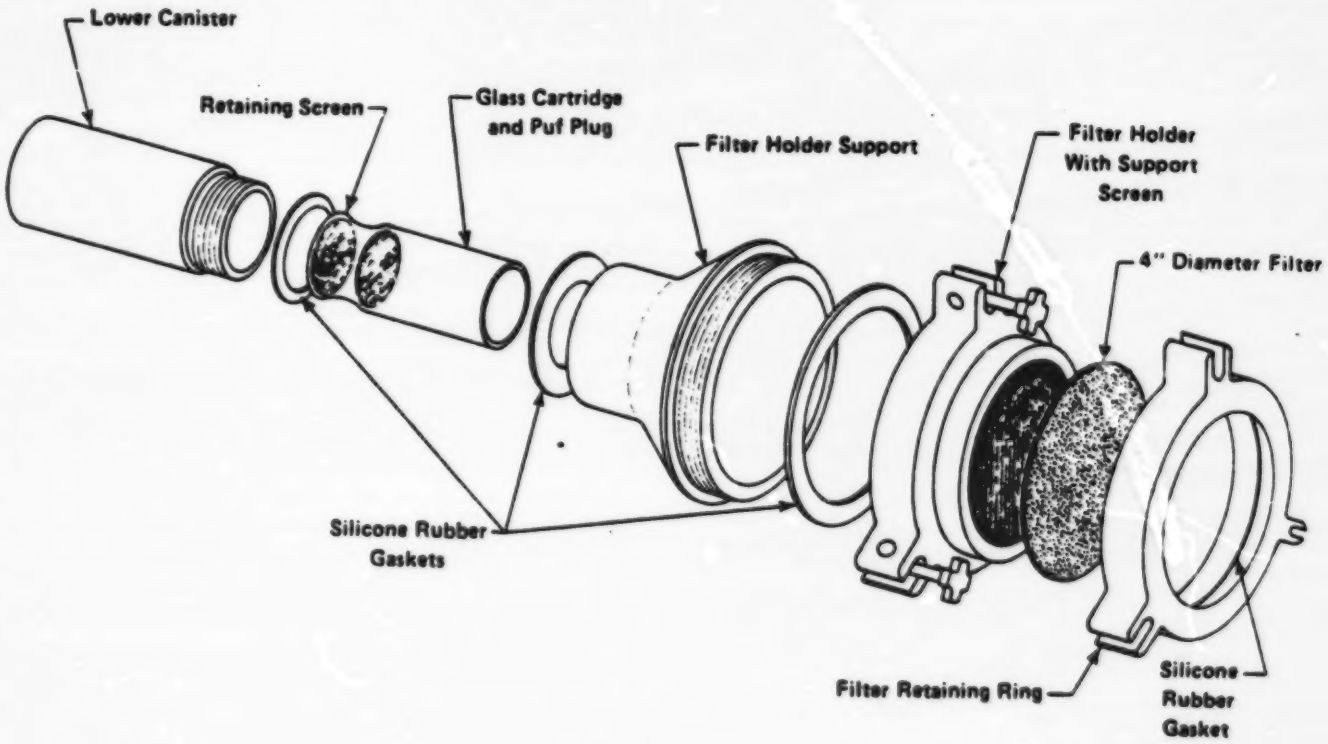


Figure 2 Sampling Module

AMBIENT AIR MONITORING
STANDARD OPERATING PROCEDURES

NUMBER: FO-051
DATE: Jan. 2, 1985
PAGE: 1 of 10
Revision No.: 2

TITLE: Operation of the GMW Model PS-1 Air Sampler

1. Purpose:

   To provide for the operation of the GMW Model PS-1 air sampler in order to collect samples representative of ambient air quality.

2. Applicability:

   This procedure is applicable to the on-site operation of the PS-1 sampler.

3. Responsibilities:

   Personnel involved in operating and maintaining the PS-1 sampler will be knowledgeable of this SOP.

4. References:

   4.1 Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA-600/4-84-041, April 1984.
   4.2 Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples, EPA-600/8-80-038, June 1980.

5. Equipment:

   5.1 GMW Model PS-1 air sampler.

6. Procedures:

   6.1 Pre-sampling activities:

   NOTE: Refer to figures 1 and 2 for identification of the sampler components.

   1. Open the PS-1 sampler hood and secure it to the back latch. Also open the sampler door.
   2. Check the meter zero on the sampler's magnehelic gauge and adjust to zero if necessary.
   3. Remove the polyethylene cover from the aluminum sampling module, then disconnect the module from the sampler's pneumatic line.
   4. Close and secure the sampler's hood and door.

5. Take the sampling module to a favorable work area, preferably an indoor location. An ice chest containing two clean sample cartridges (loaded with PUF) in their protective containers, two glass fiber filters in aluminum pouches, and a container with extra n-hexane rinsed aluminum foil should be located in the work area. The ice chest should not be cooled with ice packs at this time.
6. Unscrew and remove the sampling module's lower canister from the filter holder support.
7. Check the module's filter holder/filter holder support connection and hand tighten if necessary.
8. Check the gasket in the bottom of the lower canister, and in the base of the filter holder. Replace the gaskets if necessary.

CAUTION: Cover each hand with an unused, disposable polyethylene glove. If the gloves are not available, contact the Springfield headquarters before proceeding.

9. Open a protective container and remove the sample cartridge.
10. Unwrap the hexane rinsed aluminum foil from around the cartridge. Avoid tearing the foil as it will be needed at the end of the sampling period.
11. Slide the cartridge into the sampling module's lower canister. The end with the metal screen must be inserted first. Also note the number inscribed on the cartridge.
12. Neatly fold the aluminum foil wrapper and return it to the cartridge's protective container, then reseal the container.
13. Reconnect the sampling module's lower canister to the filter holder support. Do not attempt to over-tighten the connection since two much force will break the glass sample cartridge.
14. Record the sample cartridge number on the PS-1 Sample Information Form in the area marked "Sample Cartridge Number", see Attachment A.
15. The extra sample cartridge is designated as a trip blank cartridge. Remove the trip blank cartridge from its protective container, then unwrap the hexane rinsed aluminum foil from around the cartridge. Avoid tearing the foil as it will be needed at the end of the sampling period.
16. Note the number inscribed on the trip blank cartridge.
17. Wave the trip blank cartridge back and forth a few times, then return it to its container. Neatly fold the cartridge's aluminum foil wrapper and insert it between the cartridge and the inside of the protective container. Reseal the container.

18. Record the trip blank cartridge number on the PS-1 Sample Information Form in the area marked "Blank Cartridge Number", see Attachment A.
19. Open an aluminum pouch containing one of the clean glass fiber filters. Avoid tearing the pouch as it will be reused.
20. Wave the glass fiber filter back and forth a few times, then reinsert it back into its pouch. Reseal the pouch and place it in the ice chest. This filter is now designated as the trip blank filter.
21. Take the sampling module and the remaining unopened pouch containing the sample filter to the PS-1 sampler. You will also need a single-holed resistance plate and a silicone gasket.
22. Open the PS-1 sampler hood and secure it to the back latch. Also open the sampler's door.
23. Connect the sampling module to the samplers pneumatic line by applying torque only to the module's lower canister. Hand tighten only.
24. Remove the filter retaining ring from the module's filter holder. Place the silicone gasket on the filter holder, then position the single-holed resistance plate on top of the gasket. Place the filter retaining ring over the resistance plate and secure it to the filter holder by tightening the three swing bolts.
25. Record the elapsed time meter reading as the initial reading, then engage the sampler's power switch.
26. Plug the opening on the resistance plate with a finger. The sampler's magnehelic gauge should read exactly zero.

NOTE: If the magnehelic reading is above zero, then an air leak is present. Eliminate the leak before continuing. If the magnehelic display is below zero, then contact the Springfield headquarters before proceeding. Record all actions on the PS-1 Sample Information Form.

27. Upon completion of the leak check, disengage the sampler's power switch.
28. Remove the filter retaining ring, resistance plate, and gasket.
29. Open the aluminum pouch containing the clean glass fiber filter and remove the filter. The pouch may be discarded.
30. Center the filter, rougher side up, on the filter holder. Position the filter retaining ring over the filter and secure it to the holder by tightening the three swing bolts. Do not overtighten as the filter tends to adhere to the retaining ring.

31. Discard the used polyethylene gloves.
32. Engage the sampler's power switch and adjust the ball valve until the magnehelic gauge is near a value of 40.
33. Allow the sampler's motor to warm up for five minutes, then adjust the ball valve until the magnehelic gauge is at the value of 40 (or some other value as determined by the Springfield headquarters). A gauge value at 40 corresponds to a flowrate near 8 tcfm (theoretical cubic feet per minute).
34. Disengage the power switch and set the Paragon timer wheel to the current day and time. Also position the timer's trip pins to activate and deactivate sampling at the designated times.

NOTE: Under no circumstances may the sample cartridge be installed in the sampler for longer than 12 hours prior to the start of sampling.

35. Close and secure the sampler's hood and door.
36. Record all relevant data on the PS-1 Sample Information Form. Retain the form in a secure location.

6.2 Post-sampling activities:

NOTE: The exposed filter and cartridge must be retrieved within 6 hours after the sampling period ends.

1. Open the sampler's hood and secure it to the back latch. Also open the sampler's door.
2. Engage the sampler's power switch and allow the motor to warm up for 5 minutes, then record the magnehelic gauge display as the final reading.
3. Disengage the sampler's power switch and record the final elapsed time meter reading.
4. Disconnect the aluminum sampling module from the sampler's pneumatic line.
5. Close and secure the sampler's hood and door.
6. Take the sampling module to a favorable work area, preferably an indoor location. A cooled ice chest containing the trip blank cartridge and filter, the sample cartridge's protective container, and a container with extra hexane rinsed aluminum foil should be located in the work area.

CAUTION: Cover each hand with an unused, disposable polyethylene glove. If the gloves are not available, contact the Springfield headquarters before proceeding.

7. Unscrew and remove the sampling module's lower canister from the filter holder support.
8. Open the sample cartridge's protective container, remove the aluminum foil wrapper and unfold it.
9. Slide the cartridge out of the sampling module's lower canister.
10. Rewrap the exposed cartridge with the foil and insert it into the protective container.
11. Open the container with the spare aluminum foil, remove a piece and unfold it.
12. Carefully remove the exposed glass fiber filter from the sampling module.
13. Fold the filter in half, with the exposed surface on the inside of the fold, then fold the filter in half again.
14. Wrap the folded filter with the spare aluminum foil, then place the filter in the sample cartridge container. Reseal the cartridge container.
15. Remove the trip blank cartridge from its container and wave it back and forth a few times. Rewrap the cartridge in its original foil cover and return the cartridge to its container.
16. Remove the trip blank filter from its pouch and wave it back and forth a few times. Discard the pouch.
17. Fold the filter in half twice, then wrap it with a piece of spare aluminum foil. Place the filter in the trip blank cartridge container. Reseal the trip blank and spare aluminum foil containers.
18. Discard the used polyethylene gloves.
19. Fill out sample labels (see Attachment B) for the sample and trip blank. Stick the labels on the appropriate protective container, then return the containers to the cooled ice chest.
20. Obtain a clean, unused cloth rag and dampen it with GC grade n-hexane. Thoroughly scrub down the sampling module with the hexane moistened cloth. Then clean the sampling shelter's upper platen and inside walls with the same rag. Discard the used cloth.
21. Re-assemble the sampling module. Return the module to the PS-1, open the sampler's hood, then reconnect the module to the sampler's pneumatic line.
22. Cover the sampling module with a polyethylene bag.
23. Close and secure the sampler's hood.
24. Complete the PS-1 Sample Information Form in triplicate. Retain the original with the sample and trip blank, forward one copy to the Springfield headquarters, and file the remaining copy.
25. Repack the sample and trip blank protective containers in a cooled shipping container. Mail the cooler to the designated analytical laboratory within 24 hours after the end of sampling.

JB:bjh/sp/1913D/1,5

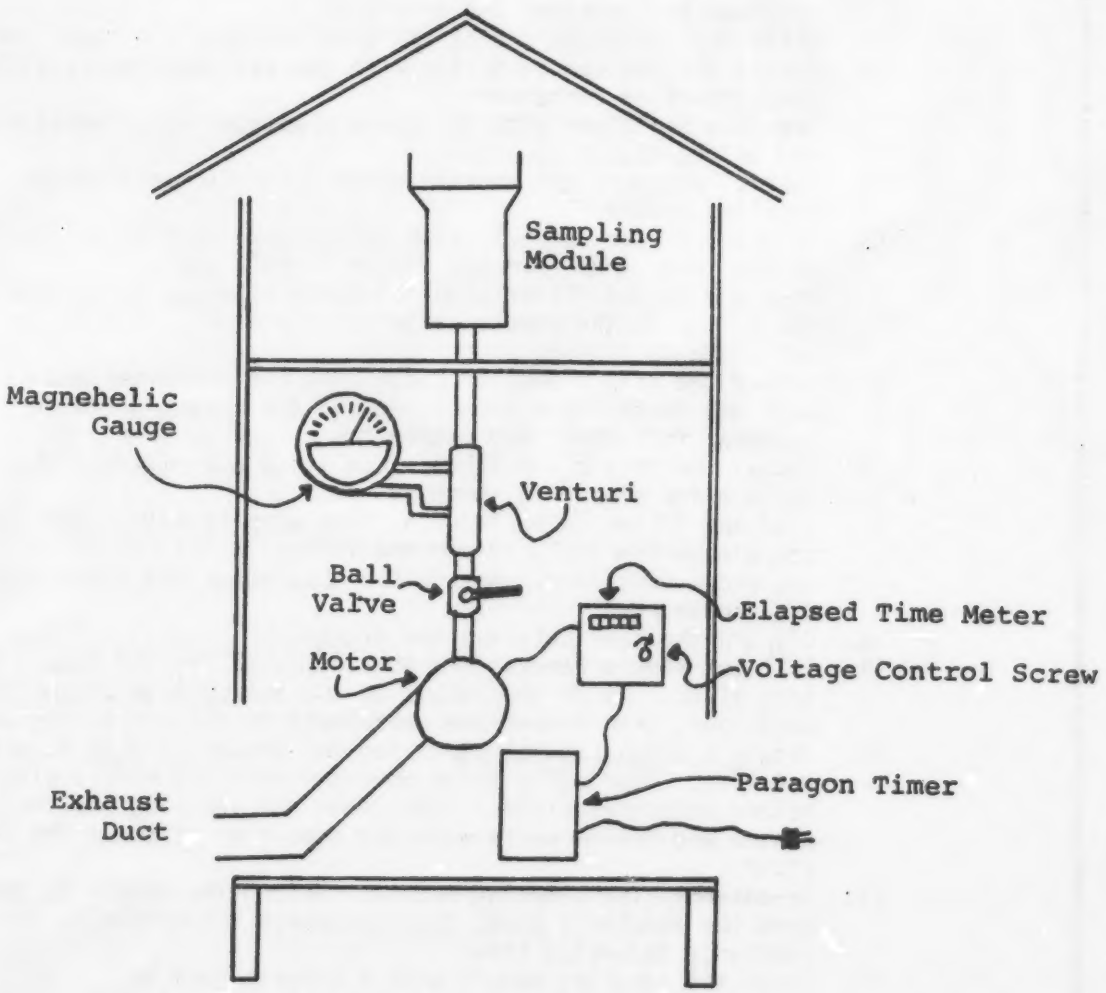


Figure 1 GMW Model PS-1 Sampler

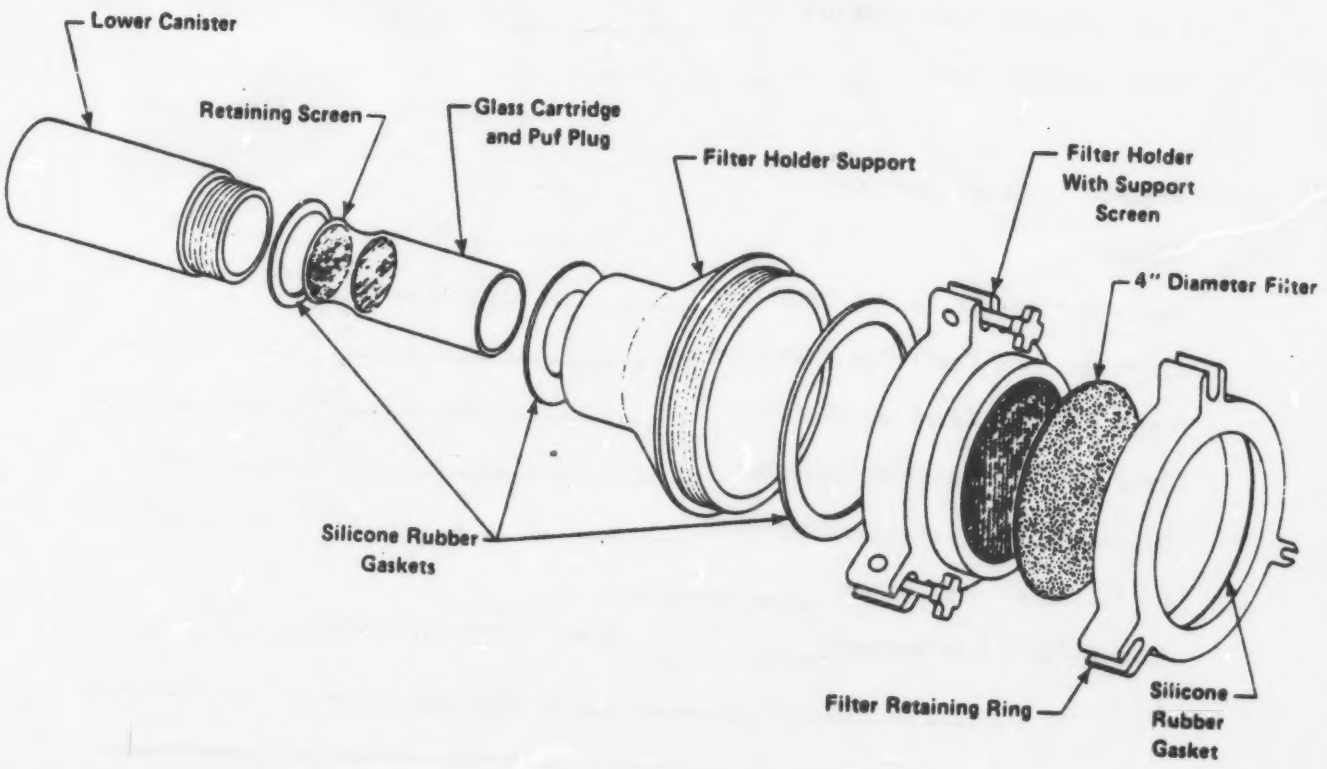


Figure 2 Sampling Module

Illinois Environmental
Protection Agency

Division of Air
Pollution Control

PS-1 Sample Information Form

Sample Number:_____ Collector:_____

Sample Location:_____

Initial Elapsed Timer Reading: ☐☐☐☐☐ ☐

Final Elapsed Timer Reading: ☐☐☐☐☐ ☐

Total Elapsed Time:_____ ___min

	Initial	Final
Magnehelic Gauge Reading:	_____	_____
Flowrate:	_____tcfm	_____tcfm

Average Flowrate During Sampling:_____tcfm

Date and Time Cartridge Installed:_____ A.M. P.M. ___/___/___

Date and Time Sampling Started: _____ A.M. P.M. ___/___/___

Date and Time Sampling Stopped: _____ A.M. P.M. ___/___/___

Date and Time Cartridge Removed: _____ A.M. P.M. ___/___/___

PS-1 Sampler IEPA Number:_____

Sample Cartridge Number:_____ Blank Cartridge Number:_____

Comments:_____

_____

For Laboratory Use

_____

_____

_____

_____

_____

CHAIN OF CUSTODY RECORD

DATE	TIME	ACTION TAKEN AND SIGNATURE

BH:rd3142C/3-4

ILLINOIS ENVIRONMENTAL PROTECTION AGENCY

SAMPLE NO. _____

CARTRIDGE NO. _____ Collector _____

COMMENT _____

APPENDIX E

USEPA Method 608;
Gas Chromatography with Electron Capture Detection

United States
Environmental Protection
Agency

Environmental Monitoring and
Support Laboratory
Cincinnati OH 45268

Research and Development

EPA

Test Method

Organochlorine Pesticides and PCBs — Method 608

1. Scope and Application

1.1 This method covers the determination of certain organochlorine pesticides and PCBs. The following parameters can be determined by this method:

Parameter	STORET No.	CAS No.
Aldrin	39330	309-00-2
α-BHC	39337	319-84-6
β-BHC	39338	319-85-7
δ-BHC	34259	319-86-8
γ-BHC	39340	58-89-9
Chlordane	39350	57-74-9
4,4'-DDD	39310	72-54-8
4,4'-DDE	39320	72-55-9
4,4'-DDT	39300	50-29-3
Dieldrin	39380	60-57-1
Endosulfan I	34361	959-98-8
Endosulfan II	34356	33212-65-9
Endosulfan sulfate	34351	1031-07-8
Endrin	39390	72-20-8
Endrin aldehyde	34366	7421-93-4
Heptachlor	39410	76-44-8
Heptachlor epoxide	39420	1024-57-3
Toxaphene	39400	8001-35-2
PCB-1016	34671	12674-11-2
PCB-1221	39488	11104-28-2
PCB-1232	39492	11141-16-5
PCB-1242	39496	53469-21-9
PCB-1248	39500	12672-29-6
PCB-1254	39504	11097-69-1
PCB-1260	39508	11096-82-5

1.2 This is a gas chromatographic (GC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 625 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and

quantitative confirmation of results for all of the parameters listed above, using the extract produced by this method.

1.3 The method detection limit (MDL, defined in Section 14.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 The sample extraction and concentration steps in this method are essentially the same as in methods 606, 609, 611 and 612. Thus, a single sample may be extracted to measure the parameters included in the scope of each of these methods. When cleanup is required, the concentration levels must be high enough to permit selection of aliquots as necessary to apply appropriate cleanup procedures. The analyst is allowed the latitude to select gas chromatographic conditions appropriate for the simultaneous measurement of combinations of these parameters.

1.5 Any modification of this method, beyond those expressly permitted, shall be considered as major modifications subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatography and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 A measured volume of sample, approximately one-liter, is solvent extracted with methylene chloride using a separatory funnel. The methylene chloride extract is dried and exchanged to hexane, during concentration to a final volume of 10 mL or less. Gas chromatographic conditions are described which permit the separation and measurement of the parameters in the extract by electron capture GC[2].

2.2 The method provides a Florisil column procedure and elemental sulfur removal procedure to aid in the elimination of interferences that may be encountered.

3. Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.5.

3.1.1 Glassware must be scrupulously cleaned[3]. Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. This should be followed by detergent washing with hot water, and rinses with tap water and distilled water. It should then be drained dry and heated in a muffle furnace at 400 °C for 15 to 30 minutes. Some thermally stable materials, such as PCBs, may not be eliminated by this treatment. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Thorough rinsing with such solvents usually eliminates PCB interference. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Interferences by phthalate esters can pose a major problem in pesticide analysis when using the elution capture detector. These compounds generally appear in the chromatogram as large eluting peaks, especially in the 15 and 50% fractions from Florisil. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Cross contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent wetted surfaces are handled. Interferences from phthalates can best be minimized by avoiding the use of plastics in the laboratory. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination[4,5]. The interferences from phthalate esters can be avoided by using a microcoulometric or electrolytic conductivity detector.

3.3 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. The cleanup procedures in Section 11 can be used to overcome many of these interferences, but unique samples may require additional cleanup approaches to achieve the MDL listed in Table 1.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[6-8] for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: 4,4'-DDT, 4,4'-DDD, the BHCs, and the PCBs. Primary standards of these toxic compounds should be prepared in a hood.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete or composite sampling.

5.1.1 Grab sample bottle—Amber glass, one-liter or one-quart volume, fitted with screw caps lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The container must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional)—Must incorporate glass sample containers for the collection of a minimum of 250 mL. Sample containers must be kept refrigerated at 4 °C and protected from light during compositing. If the sampler uses a peristaltic pump, a minimum length of compressible

silicone rubber tubing may be used. Before use, however, the compressible tubing should be thoroughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating flow meter is required to collect flow proportional composites.

5.2 Glassware (All specifications are suggested. Catalog numbers are included for illustration only).

5.2.1 Separatory funnel—2000-mL, with Teflon stopcock.

5.2.2 Drying column—Chromatographic column approximately 400 mm long × 19 mm ID, with coarse frit.

5.2.3 Chromatographic column—Pyrex, 400 mm long × 22 mm ID, with coarse fritted plate and Teflon stopcock (Kontes K-42054 or equivalent).

5.2.4 Concentrator tube, Kuderna-Danish—10-mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

5.2.5 Evaporative flask, Kuderna-Danish—500-mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs.

5.2.6 Snyder column, Kuderna-Danish—three-ball macro (Kontes K-503000-0121 or equivalent).

5.2.7 Vials—Amber glass, 10- to 15-mL capacity, with Teflon-lined screw cap.

5.3 Boiling chips—approximately 10/40 mesh. Heat to 400 °C for 30 minutes or Soxhlet extract with methylene chloride.

5.4 Water bath—Heated, with concentric ring cover, capable of temperature control (± 2 °C). The bath should be used in a hood.

5.5 Balance—Analytical, capable of accurately weighing 0.0001 g.

5.6 Gas chromatograph—An analytical system complete with gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.6.1 Column 1—1.8 m long × 4 mm ID glass, packed with 1.5% SP-2250/1.95% SP-2401 on Supelcoport (100/120 mesh) or equivalent. Column 1 was used to develop the method performance statements in Section 14. Guidelines for the use of alternate column packings are provided in Section 12.1.

5.6.2 Column 2—1.8 m long × 4 mm ID glass, packed with 3% OV-1 on Supelcoport (100/120 mesh) or equivalent.

5.6.3 Detector—Electron capture. This detector has proven effective in the analysis of wastewaters for the parameters listed in the scope, and was used to develop the method performance statements in Section 14. Guidelines for the use of alternate detectors are provided in Section 12.1.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of each parameter of interest.

6.2 Sodium hydroxide solution (10 N)—(ACS). Dissolve 40g NaOH in reagent water and dilute to 100 mL.

6.3 Sodium thiosulfate—(ACS). Granular.

6.4 Sulfuric acid solution (1 + 1)—(ACS). Slowly, add 50 mL H_2SO_4 (sp. gr. 1.84) to 50 mL of reagent water.

6.5 Acetone, hexane, isooctane (2,2,4-trimethylpentane), methylene chloride—Pesticide quality or equivalent.

6.6 Ethyl ether—Pesticide quality or equivalent, redistilled in glass if necessary.

6.6.1 Must be free of peroxides as indicated by EM Laboratories Quant test strips (Available from Scientific Products Co., Cat. No. P1126-8, and others suppliers.)

6.6.2 Procedures recommended for removal of peroxides are provided with the test strips. After cleanup, 20 mL ethyl alcohol preservative must be added to each liter of ether.

6.7 Sodium sulfate—(ACS) Granular, anhydrous. Purify by heating at 400 °C for 4 hours in a shallow tray.

6.8 Florisil—PR grade (60/100 mesh); purchase activated at 1250 °F and store in dark in glass containers with glass stoppers or foil-lined screw caps. Before use, activate each batch at least 16 hours at 130 °C in a foil covered glass container.

6.9 Mercury—Triple distilled.

6.10 Copper powder—Activated.

6.11 Stock standard solutions (1.00 µg/µL)—Stock standard solutions can be prepared from pure standard materials or purchased as certified solutions.

6.11.1 Prepare stock standard solutions by accurately weighing about 0.0100 grams of pure material. Dissolve the material in isooctane, dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. If compound purity is certified at 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.11.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4 °C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. Quality control check standards that can be used to determine the accuracy of calibration standards will be available from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

6.11.3 Stock standard solutions must be replaced after six months, or sooner if comparison with check standards indicate a problem.

7. Calibration

7.1 Establish gas chromatographic operating parameters which produce retention times equivalent to those indicated in Table 1. The gas chromatographic system may be calibrated using the external standard technique (Section 7.2) or the internal standard technique (Section 7.3).

7.2 External standard calibration procedure:

7.2.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with isooctane. One of the external standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.2.2 Using injections of 2 to 5 µL of each calibration standard, tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to amount injected (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.2.3 The working calibration curve or calibration factor must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±10%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve or calibration factor must be prepared for that compound.

7.3 Internal standard calibration procedure. To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask. To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with isooctane. One of the standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.3.2 Using injections of 2 to 5 µL of each calibration standard, tabulate peak height or area responses against concentration for each compound and internal standard, and calculate response factors (RF) for each compound using equation 1.

Eq. 1. $RF = (A_s C_{is})/(A_{is} C_s)$

where:

A_s = Response for the parameter to be measured.

A_{is} = Response for the internal standard.

C_{is} = Concentration of the internal standard, (µg/L).

C_s = Concentration of the parameter to be measured, (µg/L).

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.3.3 The working calibration curve or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±10%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve must be prepared for that compound.

7.4 The cleanup procedure in Section 11 utilizes Florisil chromatography. Florisil from different batches or sources may vary in absorptive capacity. To standardize the amount of Florisil which is used, the use of lauric acid value[9] is suggested. The referenced procedure determines the adsorption from hexane solution of lauric acid (mg) per gram Florisil. The amount of Florisil to be used for each column is calculated by dividing this factor into 110 and multiplying by 20 g.

7.5 Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from the reagents.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and the analysis of spiked samples as a continuing check on performance. The laboratory is required to maintain performance records to define the quality of data that is generated. Ongoing performance checks must be compared with established performance criteria to determine if the results of analyses are within accuracy and precision limits expected of the method.

8.1.1 Before performing any analyses, the analyst must demonstrate the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of the rapid advances that are occurring in chromatography, the analyst is permitted certain options to improve the separations or lower the cost of measurements. Each time such modifications are made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 The laboratory must spike and analyze a minimum of 10% of all samples to monitor continuing laboratory performance. This procedure is described in Section 8.4.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 Select a representative spike concentration for each compound to be measured. Using stock standards, prepare a quality control check sample concentrate in acetone 1000 times more concentrated than the selected concentrations. Quality control check sample concentrates, appropriate for use with this method, will be available from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

8.2.2 Using a pipet, add 1.00 mL of the check sample concentrate to each of a minimum of four 1000-mL aliquots of reagent water. A representative wastewater may be used in place of the reagent water, but one or more additional aliquots must be analyzed to determine background levels, and the spike level must exceed twice the background level for the test to be valid. Analyze the aliquots according to the method beginning in Section 10.

8.2.3 Calculate the average percent recovery, (R), and the standard deviation of the percent recovery (s), for the results. Wastewater background corrections must be made before R and s calculations are performed.

8.2.4 Using Table 2, note the average recovery (X) and standard deviation (p) expected for each method parameter. Compare these to the calculated values for R and s. If $s > 2p$ or $|X - R| > 2p$, review potential problem areas and repeat the test.

8.2.5 The U.S. Environmental Protection Agency plans to establish performance criteria for R and s based upon the results of interlaboratory testing. When they become available, these criteria must be met before any samples may be analyzed.

8.3 The analyst must calculate method performance criteria and define

the performance of the laboratory for each spike concentration and parameter being measured.

8.3.1 Calculate upper and lower control limits for method performance:

Upper Control Limit (UCL) = R + 3s
Lower Control Limit (LCL) = R − 3s

where R and s are calculated as in Section 8.2.3. The UCL and LCL can be used to construct control charts[10] that are useful in observing trends in performance. The control limits above be replaced by method performance criteria as they become available from the U.S. Environmental Protection Agency.

8.3.2 The laboratory must develop and maintain separate accuracy statements of laboratory performance for wastewater samples. An accuracy statement for the method is defined as R ± s. The accuracy statement should be developed by the analysis of four aliquots of wastewater as described in Section 8.2.2, followed by the calculation of R and s. Alternately, the analyst may use four wastewater data points gathered through the requirement for continuing quality control in Section 8.4. The accuracy statements should be updated regularly[10].

8.4. The laboratory is required to collect a portion of their samples in duplicate to monitor spike recoveries. The frequency of spiked sample analysis must be at least 10% of all samples or one sample per month, whichever is greater. One aliquot of the sample must be spiked and analyzed as described in Section 8.2. If the recovery for a particular parameter does not fall within the control limits for method performance, the results reported for that parameter in all samples processed as part of the same set must be qualified as described in Section 13.5. The laboratory should monitor the frequency of data so qualified to ensure that it remains at or below 5%.

8.5 Before processing any samples, the analyst should demonstrate through the analysis of a one-liter aliquot of reagent water, that all glassware and reagent interferences are under control. Each time a set of samples is extracted or there is a change in reagents, a laboratory reagent blank should be processed as a safeguard against laboratory contamination.

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to monitor the precision of the sampling technique. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should perform analysis of standard reference materials and participate in relevant performance evaluation studies.

9. Sample Collection, Preservation, and Handling

9.1 Grab samples must be collected in glass containers. Conventional sampling practices[11] should be followed, except that the bottle must not be prewashed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

9.2 The samples must be iced or refrigerated at 4 °C from the time of collection until extraction. If the samples will not be extracted within 72 hours of collection, the sample should be adjusted to a pH range of 5.0 to 9.0 with sodium hydroxide or sulfuric acid. Record the volume of acid or base used. If aldrin is to be determined, add sodium thiosulfate when residual chlorine is present. U.S. Environmental Protection Agency methods 330.4 and 330.5 may be used to measure chlorine residual[12]. Field test kits are available for this purpose.

9.3 All samples must be extracted within 7 days and completely analyzed within 40 days of extraction[2].

10. Sample Extraction

10.1 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a two-liter separatory funnel.

10.2 Add 60 mL methylene chloride to the sample bottle, seal, and shake 30 seconds to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for two minutes with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250-mL Erlenmeyer flask.

10.3 Add a second 60-mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

10.4 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the Kuderna Danish if the requirements of Section 8.2 are met.

10.5 Pour the combined extract through a drying column containing about 10 cm of anhydrous sodium sulfate, and collect the extract in the K-D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

10.6 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL methylene chloride to the top. Place the K-D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 minutes. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes.

10.7 Increase the temperature of the hot water bath to about 80 °C. Momentarily remove the Snyder column, add 50 mL of hexane and a new boiling chip and reattach the Snyder column. Prewet the column by adding about 1 mL of hexane to the top. Concentrate the solvent extract as before. The elapsed time of concentration should be 5 to 10 minutes. When the apparent volume of liquid reaches 1 mL, remove the K-D apparatus and allow it to drain and cool at least 10 minutes.

10.8 Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of hexane. A 5-mL syringe is recommended for this operation. Stopper the concentrator tube and store refrigerated if further processing will not be performed immediately. If the extracts will be stored longer than two days, they should be transferred to Teflon-sealed screw-cap bottles. If the sample extract requires no further cleanup, proceed with gas chromatographic analysis. If the sample requires cleanup proceed to Section 11.

10.9 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

11. Cleanup and Separation

11.1 Cleanup procedures may not be necessary for a relatively clean sample matrix. The cleanup procedures recommended in this method have been used for the analysis of various clean waters and industrial effluents. If particular circumstances demand the use of an alternative cleanup procedure, the analyst must determine the elution profile and demonstrate that the recovery of each compound of interest is no less than 85%. The Florisil column allows for a select fractionation of the compounds and will eliminate polar materials. Elemental sulfur interferes with the electron capture gas chromatography of certain pesticides, but can be removed by the techniques described below.

11.2 Florisil column cleanup:

11.2.1 Add a weight of Florisil (nominally 21 g) predetermined by calibration (Section 7.4 and 7.5), to a chromatographic column. Settle the Florisil by tapping the column. Add sodium sulfate to the top of the Florisil to form a layer 1 to 2 cm deep. Add 60 mL of hexane to wet and rinse the sodium sulfate and Florisil. Just prior to exposure of the sodium sulfate to air, stop the elution of the hexane by closing the stopcock on the chromatography column. Discard the eluate.

11.2.2 Adjust the sample extract volume to 10 mL with hexane and transfer it from the K-D concentrator tube to the Florisil column. Rinse the tube twice with 1 to 2 mL hexane, adding each rinse to the column.

11.2.3 Place a 500-mL K-D flask and clean concentrator tube under the chromatography column. Drain the column into the flask until the sodium sulfate layer is nearly exposed. Elute the column with 200 mL of 6% ethyl ether in hexane (V/V) (Fraction 1) using a drip rate of about 5 mL/min. Remove the K-D flask and set aside for later concentration. Elute the column again, using 200 mL of 15% ethyl ether in hexane (V/V)(Fraction 2), into a second K-D flask. Perform the third elution using 200 mL of 50% ethyl ether in hexane (V/V)(Fraction 3). The elution patterns for the pesticides an PCB's are shown in Table 2.

11.2.4 Concentrate the eluates by standard K-D techniques (Section 10.6), substituting hexane for the glassware rinses and using the water bath at about 85 °C. Adjust final volume to 10 mL with hexane. Analyze by gas chromatography.

11.3 Elemental sulfur will usually elute entirely in Fraction 1 of the Florisil column cleanup. To remove sulfur interference from this fraction or the original extract, pipet 1.00 mL of the concentrated extract into a clean concentrator tube or Teflon-sealed vial. Add one to three drops of mercury and seal[13]. Agitate the contents of the vial for 15 to 30 seconds. Prolonged shaking (two hours) may be required. If so, this may be accomplished with a reciprocal shaker. Alternatively, activated copper powder may be used for sulfur removal[14]. Analyze by gas chromatography.

12. Gas Chromatography

12.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. This table includes retention times and MDL that were obtained under these conditions. Examples of the parameter separations achieved by column 1 are shown in Figures 1 to 10. Other packed columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met. Capillary (open-tubular) columns may also be used if the relative standard deviations of responses for replicate injections are demonstrated to be less than 6% and the requirements of Section 8.2 are met.

12.2 Calibrate the system daily as described in Section 7.

12.3 If the internal standard approach is being used, the internal standard must be added to the sample extract and mixed thoroughly immediately, before injection into the instrument.

12.4 Inject 2 to 5 µL of the sample extract using the solvent-flush technique[15]. Smaller (1.0 µL) volumes can be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 µL, the total extract volume, and the resulting peak size in area or peak height units.

12.5 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

12.6 If the response for the peak exceeds the working range of the system, dilute the extract and reanalyze.

12.7 If the measurement of the peak response is prevented by the presence of interferences, further cleanup is required.

13. Calculations

13.1 Determine the concentration of individual compounds in the sample.

13.1.1 If the external standard calibration procedure is used, calculate the amount of material injected from the peak response using the calibration curve or calibration factor in Section 7.2.2. The concentration in the sample can be calculated from equation 2:

$$\text{Eq. 2. Concentration, } \mu g/L = \frac{(A)(V_t)}{(V_i)(V_s)}$$

where:

- A = Amount of material injected, in nanograms.
- V_i = Volume of extract injected (µL).
- V_t = Volume of total extract (µL).
- V_s = Volume of water extracted (mL).

13.1.2 If the internal standard calibration procedure was used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.3.2 and equation 3.

Eq. 3

$$\text{Concentration, } \mu g/L = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)}$$

where:

- A_s = Response for the parameter to be measured.
- A_{is} = Response for the internal standard.
- I_s = Amount of internal standard added to each extract (µg).
- V_o = Volume of water extracted, in liters.

13.2 When it is apparent that two or more PCB (Aroclor) mixtures are present, the Webb and McCall procedure[16] may be used to identify and quantify the Aroclors.

13.3 For multicomponent mixtures (chlordane, toxaphene and PCBs) match retention times of peaks in the standards with peaks in the sample. Quantitate every identifiable peak unless interference with individual peaks persist after cleanup. Add peak height or peak area of each identified peak in the chromatogram. Calculate as total response in the sample versus total response in the standard.

13.4 Report results in micrograms per liter without correction for recovery data. When duplicate and spiked samples are analyzed, report all data obtained with the sample results.

13.5 For samples processed as part of a set where the laboratory spiked sample recovery falls outside of the control limits in Section 8.3, data for the affected parameters must be labeled as suspect.

14. Method Performance

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero[1]. The MDL concentrations listed in Table 1 were obtained using reagent water[17]. Similar results were achieved using representative wastewaters.

14.2 This method has been tested for linearity of spike recovery from reagent water and has been demonstrated to be applicable over the concentration range from 4 × MDL up to 1000 × MDL with the following exceptions: Chlordane recovery at 4 × MDL was low (60%); Toxaphene recovery was demonstrated linear over the range of 10 × MDL to 1000 × MDL[17].

14.3 In a single laboratory (Southwest Research Institute), using spiked wastewater samples, the average recoveries presented in Table 3 were obtained[4]. Each spiked sample was analyzed in triplicate on two separate days. The standard deviation of the percent recovery is also included in Table 3.

14.4 The U.S. Environmental Protection Agency is in the process of conducting an interlaboratory method study to fully define the performance of this method.

References

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3. ASTM Annual Book of Standards, Part 31, D3694, "Standard Practice for Preparation of Sample Containers and for Preservation," American Society for Testing and Materials, Philadelphia, PA, p. 679, 1980.
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5. Giam, C.S., Chan, H.S., "Control of Blanks in the Analysis of Phthalates in Air and Ocean Biota Samples," U.S. National Bureau of Standards, Special Publication 442, pp. 701-708, 1976.
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15. Burke, J.A., "Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects," *Journal of the Association of Official Analytical Chemists, 48,* 1037 (1965).
16. Webb, R.G., and McCall, A.C., "Quantitative PCB Standards for Electron Capture Gas Chromatography," *Journal of Chromatographic Science, 11,* 366 (1973).
17. "Method Detection Limit and Analytical Curve Studies, EPA Methods 606, 607, and 608," Special letter report for EPA Contract 68-03-2606. Environmental Monitoring and Support Laboratory—Cincinnati, Ohio 45268.

Table 1. *Chromatographic Conditions and Method Detection Limits*

Parameter	Retention Time (min.) Column 1	Column 2	Method Detection Limit µg/L
α-BHC	1.35	1.82	0.003
γ-BHC	.70	2.13	0.004
β-BHC	1.90	1.97	0.006
Heptachlor	2.00	3.35	0.003
δ-BHC	2.15	2.20	0.009
Aldrin	2.40	4.10	0.004
Hepachlor epoxide	3.50	5.00	0.083
Endosulfan I	4.50	6.20	0.014
4,4'-DDE	5.13	7.15	0.004
Dieldrin	5.45	7.23	0.002
Endrin	6.55	8.10	0.006
4,4'-DDD	7.83	9.08	0.011
Endosulfan II	8.00	8.28	0.004
4,4'-DDT	9.40	11.75	0.012
Endrin aldehyde	11.82	9.30	0.023
Endosulfan sulfate	14.22	10.70	0.066
Chlordane	mr	mr	0.014
Toxaphene	mr	mr	0.24
PCB-1016	mr	mr	nd
PCB-1221	mr	mr	nd
PCB-1232	mr	mr	nd
PCB-1242	mr	mr	0.065
PCB-1248	mr	mr	nd
PCB-1254	mr	mr	nd
PCB-1260	mr	mr	nd

Column 1 conditions: Supelcoport (100/120 mesh) coated with 1.5% SP-2250/1.95% SP-2401 packed in a 1.8 m long × 4 mm ID glass column with 5% Methane/95% Argon carrier gas at a flow rate of 60 mL/min. Column temperature isothermal at 200°C, except for PCB-1016 through PCB-1248, which should be measured at 160°C.

Column 2 conditions: Supelcoport (100/120 mesh) coated with 3% OV-1 in a 1.8 m long × 4 mm ID glass column with 5% Methane/95% Argon carrier gas at a flow rate of 60 mL/min. Column temperature, isothermal at 200°C, for the pesticides; 140°C for PCB-1221 and 1232; 170°C for PCB-1016 and 1242 to 1268.

mr — Multiple peak response. See Figures 2 thru 10.

nd — Not determined.

Table 2. *Distribution of Chlorinated Pesticides and PCBs into Florisil Column Fractions[2]*

Parameter	Percent Recovery by Fraction: Fraction 1	Fraction 2	Fraction 3
Aldrin	100		
α-BHC	100		
β-BHC	97		
δ-BHC	98		
γ-BHC	100		
Chlordane	100		
4,4'-DDD	99		
4,4'-DDE	98		
4,4'-DDT	100		
Dieldrin	0	100	
Endosulfan I	37	64	
Endosulfan	0	7	91
Endosulfan sulfate	0	0	106
Endrin	4	96	
Endrin aldehyde	0	68	26
Heptachlor	100		
Heptachlor epoxide	100		
Toxaphene	96		
PCB-1016	97		
PCB-1221	97		
PCB-1232	95	4	
PCB-1242	97		
PCB-1248	103		
PCB-1254	90		
PCB-1260	95		

Eluant composition by fraction:
Fraction 1 — 6% ethyl ether in hexane
Fraction 2 — 15% ethyl ether in hexane
Fraction 3 — 50% ethyl ether in hexane

Table 3. *Single Operator Accuracy and Precision*

Parameter	Average Percent Recovery	Standard Deviation %	Spike Range (µg/L)	Number of Analyses	Matrix Types
Aldrin	89	2.5	2.0	15	3
α-BHC	89	2.0	1.0	15	3
β-BHC	88	1.3	2.0	15	3
δ-BHC	86	3.4	2.0	15	3
γ-BHC	97	3.3	1.0	15	3
Chlorane	93	4.1	20	21	4
4-4'-DDD	92	1.9	6.0	15	3
4,4'-DDE	89	2.2	3.0	15	3
4,4'-DDT	92	3.2	8.0	15	3
Dieldrin	95	2.8	3.0	15	2
Endosulfan I	96	2.9	3.0	12	2
Endosulfan II	97	2.4	5.0	14	3
Endosulfan sulfate	99	4.1	15	15	3
Endrin	95	2.1	5.0	12	2
Endrin aldehyde	87	2.1	12	11	2
Heptachlor	88	3.3	1.0	12	2
Heptachlor epoxide	93	1.4	2.0	15	3
Toxaphene	95	3.8	200	18	3
PCB-1016	94	1.8	25	12	2
PCB-1221	96	4.2	55-110	12	2
PCB-1232	88	2.4	110	12	2
PCB-1242	92	2.0	28-56	12	2
PCB-1248	90	1.6	40	12	2
PCB-1254	92	3.3	40	18	3
PCB-1260	91	5.5	80	18	3

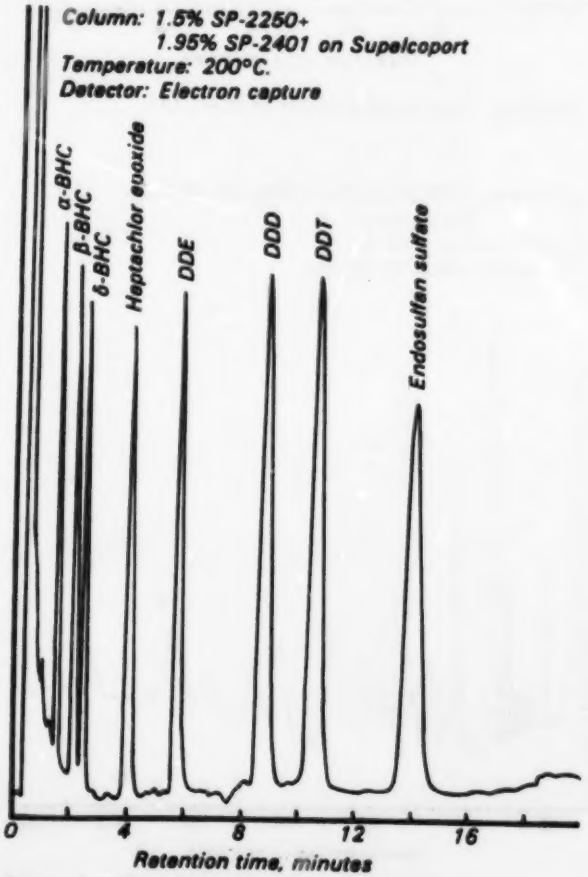

Figure 1. *Gas chromatogram of pesticides.*

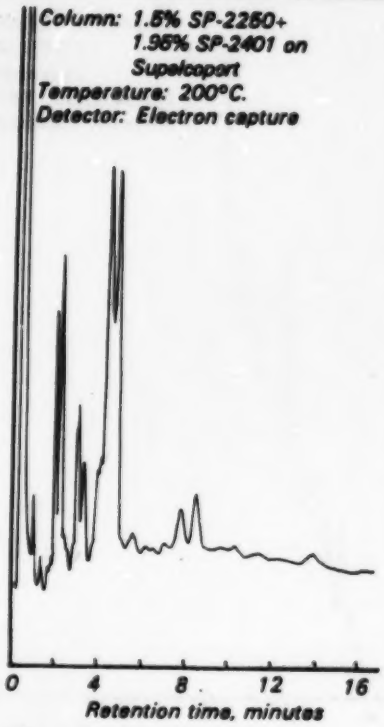

Figure 2. *Gas chromatogram of chlordane.*

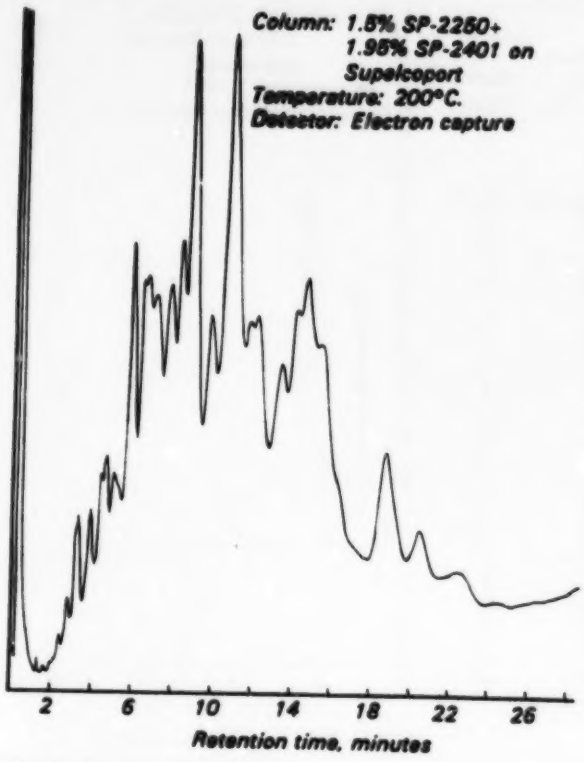

Figure 3. *Gas chromatogram of toxaphene.*

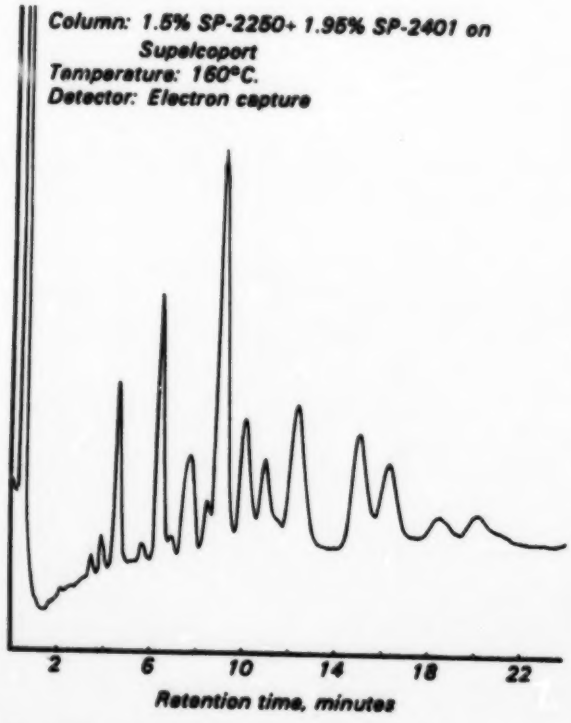

Figure 4. *Gas chromatogram of PCB-1016.*

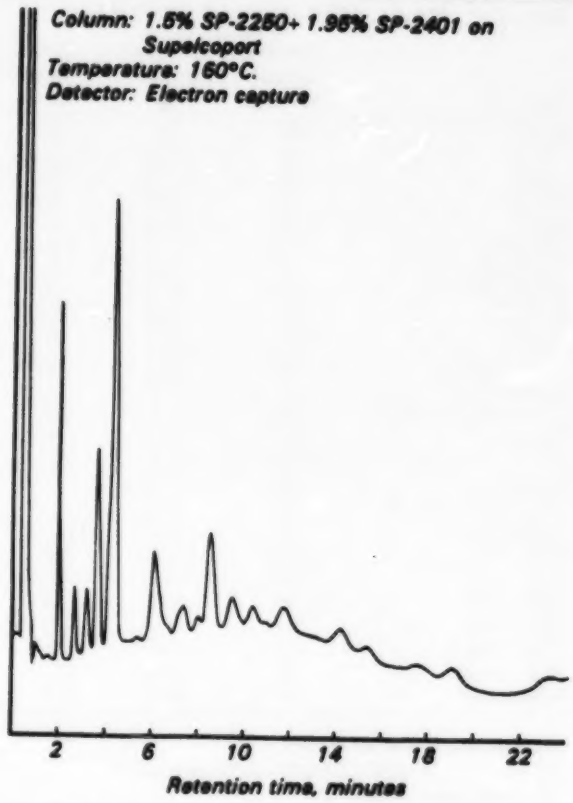

Figure 5. *Gas chromatogram of PCB-1221.*

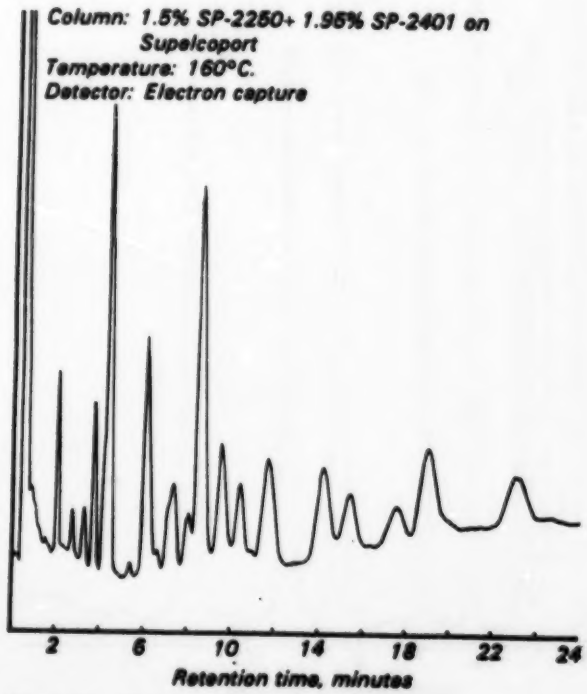

Figure 6. *Gas chromatogram of PCB-1232.*

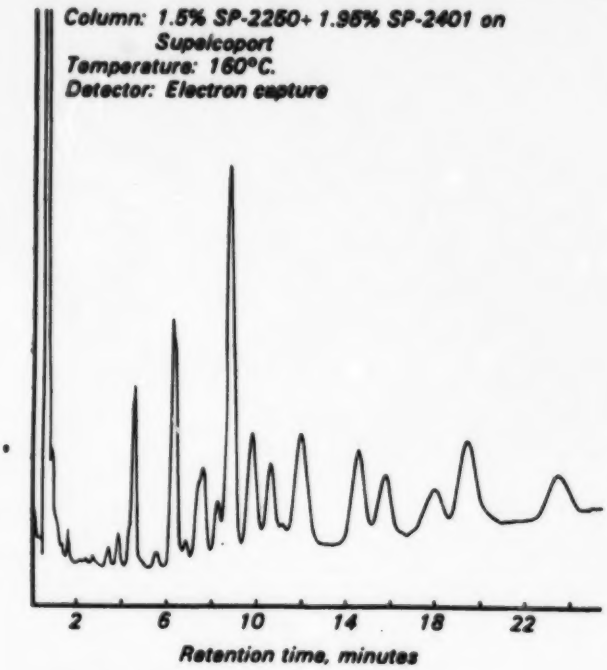

Figure 7. *Gas chromatogram of PCB-1242.*

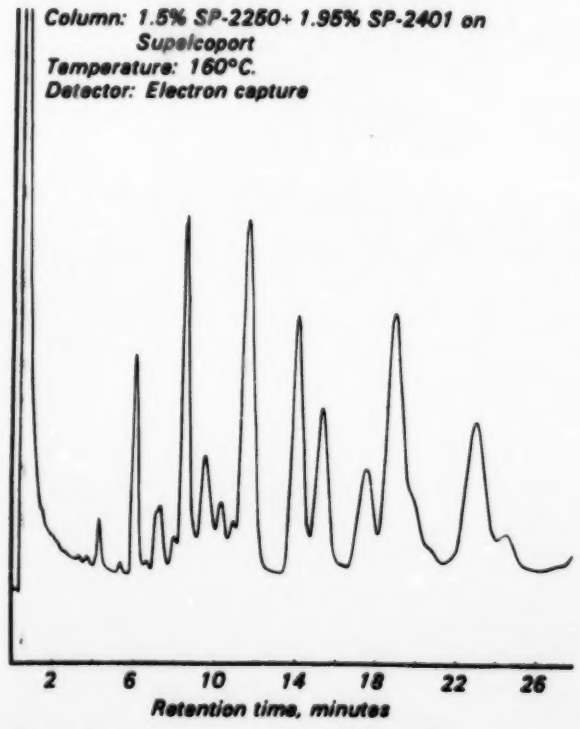

Figure 8. *Gas chromatogram of PCB-1248.*

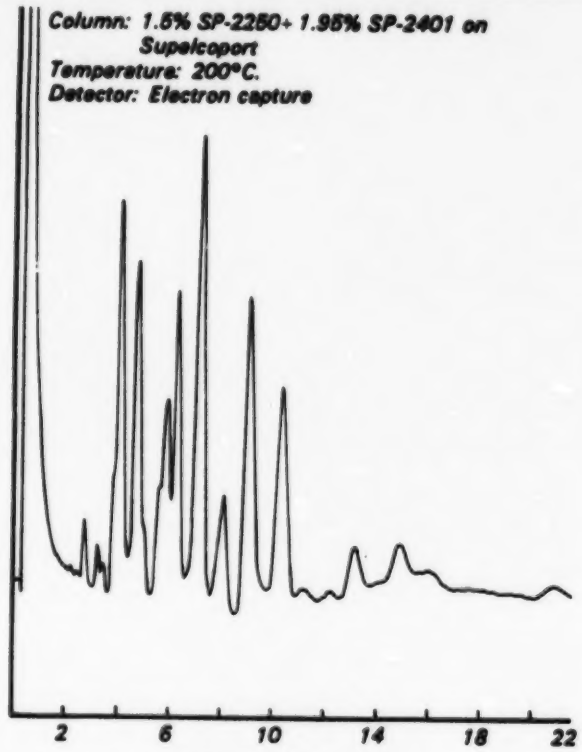

Figure 9. *Gas chromatogram of PCB-1254.*

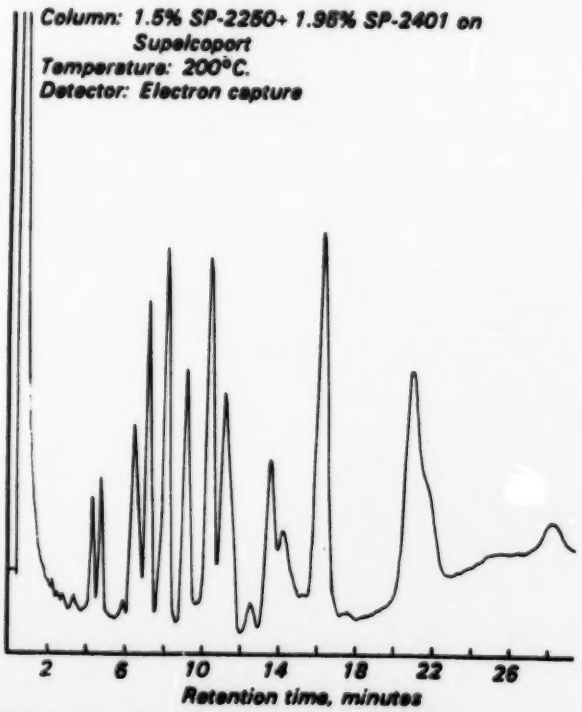

Figure 10. *Gas chromatogram of PCB-1260.*

APPENDIX F

Lake Calumet PCB Sampling Program Data Forms

RADIAN
CORPORATION

12 February 1985

RECEIVED
FEB 19 1985
IEPA - DAPC - SPFLD

Mr. James Buckert
State of Illinois EPA
2200 Churchill Road
Springfield, IL 62706

Dear Mr. Buckert:

Enclosed are copies of the information that you requested in our telephone conversation yesterday. I have included a copy of our sample log-in sheet and information from the analyst's notebook for PCB analysis.

Radian assigns each set of samples received a unique sequence number. Each sample of the set is assigned a lab code comprising of the sequence number and a numerical digit. Up to 100 samples per set can be logged in under one sequence number.

For quantitation of PCBs, Radian follows EPA Method 608 with the following modifications:

- Peak area is used
- Depending on the PCB, several peaks are used in the quantitation. For Aroclor 1242, the three largest peaks are used. A response factor based on the combined areas for the three peaks is calculated for the standard.

Although Radian has analyzed PCBs for many governmental and private clients, we have always referenced EPA Method 608 with the modifications, if applicable.

If you have any other questions or need additional information, feel free to call me.

Sincerely,

Maureen A. McDevitt

Maureen A. McDevitt
Staff Scientist

/jb

cc: Steve Copenhaver

8501 Mo-Pac Blvd. / P.O. Box 9948 / Austin, Texas 78766 / (512)454-4797

RADIAN SEQUENCE NUMBER: 4257

Key Word Sample Description: IEPA

Client: ILLINOIS EPA
Client Contact: JIM BUCKERT
Radian Contact: M. McDEVITT
Date Collected/Received: 2/4/85
Analyte(s): PCB'S
Hazard Index:
Assigned by:

Contract No.: 251-037
Phone No.:
To labs: IH/EXT/GC
No. of Samples: 2
Medium: AIR
Protection Code:
Assigned by:

Retain Samples Until:
Storage Location: IH LAB

Retain Extracts Until: 6/85
Extract Location: IH OR GC LAB

Remarks:

Custody Record:

Name	Inclusive Time/Date of Custody	Initials
Kathy Swearingen	2/4/85	KS

SAMPLE INFORMATION

FIELD CODE	LAB CODE	SAMPLE DESCRIPTION
1. SAMPLE #5; CARTRIDGE #3	4257-1	SAMPLE
2. SAMPLE #5; CARTRIDGE #5	4257-2	BLANK

Samples logged in by:KS

Date: 2/11/85

Report due:
Advance the paper so that the next line will be printed at the top of the page. Then hit return.
>

IEPA # 251-037-01 PCB 1242 Analysis

Column: M/P 6.5% SP-2250 + 1.95% SP-2401 on 100/120 Supelcoport 6 ft. x 2mm ID
Program: 160°C Isothermal
Det. Temp: 300°C ECD
Inlet: 200°C
Carrier: Argon/Methane @ 30 ml/min
Make-up: Nitrogen @ 30 ml/min

2/11/85

1)	Hexane Blank	5.0 ul	Att = 2 TH = 1	
2)	0.5 ng/ul Std. PCB 1242	5.0 ul	" "	
3)	IEPA Sample #5, Cartridge #3	5.0 ul	" "	(Sample)
4)	IEPA Sample #5, Cartridge #5	5.0 ul	" "	(Blank)
5)	IEPA Sample #4, Cartridge #8	5.0 ul	" "	(Sample)
6)	IEPA Sample #4, Cartridge #7	5.0 ul	" "	(Blank)
7)	0.1 ng/ul Std PCB 1242	5.0 ul	" "	
8)	1.0 ng/ul Std PCB 1242	5.0 ul	" "	
9)	IEPA Sample #4, Cartridge #8	5.0 ul	" "	(Sample Duplicate)

Instrument Log Book

ssed & Understood by me.	Date	Invented by	Date	
		Recorded by		

Column: M/P 1.5% SP-2250 + 1.95% SP-2401 on 100/120 Supelcoport 6 ft. x 2mm ID

Program: 160°C Isothermal

Det. Temp: 300°C ECD

Inlet: 200°C

Carrier: Argon/Methane @ 30ml/min

Make-up: Nitrogen @ 30ml/min

Standards PCB 1242 Dilution Solvent Hexane

5000 ng/ul → 1/100 dilt. = 50 ng/ul std
50 ng/ul → 1/10 dilt. = 5 ng/ul std
5 ng/ul → 1/5 dilt. = 1 ng/ul std
5 ng/ul → 1/10 dilt. = 0.5 ng/ul std
1 ng/ul → 1/10 dilt. = 0.1 ng/ul std
0.5 ng/ul → 1/10 dilt. = 0.05 ng/ul std

Spikes

5 ng/ul → 1/10 dilt. = 0.5 ng/ul
0.5 ng/ul → 1/100 dilt. = 0.005 ng/ul

0.005 ng/ul x 24 ul = 0.12 ng
0.005 ng/ul x 48 ul = 0.24 ng

} each was spiked on separate putty plugs of 5 different samplers then extracted.

Analysis

(1)	Hexane Blank	5.0 ml	
2)	IEPA Blanks (3)	5.0 ml	} Clean-up Analysis
3)	IEPA Blanks (8)	5.0 ml	
4)	IEPA Filters (6)	5.0 ml	
5)	0.12 ng Ext. Spike	5.0 ml	
6)	0.24 ng Ext. Spike	5.0 ml	
7)	0.05 ng/ml Std	5.0 ml	
8)	0.1 ng/ml Std	5.0 ml	
9)	0.5 ng/ml Std	5.0 ml	
10)	1.0 ng/ml Std	5.0 ml	
11)	1.0 ng/ml Std	5.0 ml	
12)	Hexane Blank	5.0 ml	
13)	0.12 ng Ext. Spike	5.0 ml	
14)	0.24 ng Ext. Spike	5.0 ml	
15)	Q.C. WP-678 PCB-1242 Conc. 8	5.0 ml	

```
LIST  LIST
PEAK CAPACITY: 1159

ZERO   =  5. 0.1
ATT 2↑ =  2
CHT SP =  0 5
PK WD  =  0 16
THRSH  =  1
AR REJ =  1000
```


nessed & Understood by me.	Date	Invented by	Date
		Recorded by	10/30/84

Standards Calculations

0.1 mg/ml Std (5.0 ml) = 0.5 mg

3 peaks
1) 7611
2) 30598
3) 31163
69372 area / 0.5 mg = 138744 area/mg

0.5 mg/ml Std (5.0 ml) = 2.5 mg

1) 52910
2) 79126
3) 173320
305356 area / 2.5 mg = 122142 area/mg

1.0 mg/ml Std (5.0 ml) = 5.0 mg

1) 117520
2) 171320
3) 336330
625170 area / 5.0 mg = 125034 area/mg

Avg. Mean ± Std. Dev. = 128640 ± 8869

essed & Understood by me. | Date | Invented by | Date 2/12/85
Recorded by

Book No. _____ TITLE _____

Page No. _____

Spike Calculations

0.24 ug Spike in 0.2 ml (5.0 ml)

2 peaks

1) 85778
2) 258110
343888 / 77984 $\frac{\text{Area}}{\text{ug}}$ = 4.41 / 5.0 ml x 0.2 ml (200 ml) = **0.176 ug**

(D.p) 0.24 ug Spike in 0.2 ml (5.0 ml)

1) 85519
2) 283470
368989 / 77984 = 4.73 / 5.0 l x 0.2 ml (200 ml) = **.189 ug**

0.12 ug Spike in 0.2 ml (5.0 ml)

1) 34537
2) 223720
258257 / 77984 = 3.31 / 5.0 ml x 200 ul = **0.132 ug**

0.12 ug Spike in 0.2 ml (5.0 ml)

1) 46184
2) 218060
264244 / 77984 = 3.39 / 5.0 ml x 200 ul = **0.136 ug**

Spiked 0.12 ug Recovered 0.132 ug = **110%**
0.24 ug 0.189 ug = **78.8%**

To Page No. _____

Witnessed & Understood by me. | Date | Invented by | Date 12/17/84

Recorded by

GMW MODEL PS-1 CALIBRATION FORM

Name:_____ Date:_____

Site Address:_____

PS-1 Shelter No.:_____ Station Pressure:_____

GMW Model 40' OCU No.:_____

Magnehelic Gauge Reading	Manometer Reading (in. H_2O)	OCU Flow-Rate (tcfm)	Temp. (^oC)
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

Comments:_____

_____

_____

_____

PS-1 Calibration Table

Sampler No. - 13162
Calibration Date - 07/25/84
Slope - 1.2159
Intercept - 0.0535
Std. Error - 0.1193
Corr. Coeff. - 0.9996

City _____

Location _____

Address _____

Magnehelic Reading	Flowrate (tcfm)	Magnehelic Reading	Flowrate (tcfm)	Magnehelic Reading	Flowrate (tcfm)
70	10.23	56	9.15	42	7.93
69	10.15	55	9.07	41	7.84
68	10.08	54	8.99	40	7.74
67	10.01	53	8.91	39	7.65
66	9.93	52	8.82	38	7.55
65	9.86	51	8.74	37	7.45
64	9.78	50	8.65	36	7.35
63	9.70	49	8.56	35	7.25
62	9.63	48	8.48	34	7.14
61	9.55	47	8.39	33	7.04
60	9.47	46	8.30	32	6.93
59	9.39	45	8.21	31	6.82
58	9.31	44	8.12	30	6.71
57	9.23	43	8.03	29	6.60

This calibration table is void after 01/25/85

Originator _____ Date _____

Illinois Environmental Protection Agency

Division of Air Pollution Control

PS-1 Sample Information Form

Sample Number:_____ Collector:_____

Sample Location:_____

Initial Elapsed Timer Reading: ☐☐☐☐☐ ☐

Final Elapsed Timer Reading: ☐☐☐☐☐ ☐

Total Elapsed Time:_____ ___min

	Initial	Final
Magnehelic Gauge Reading:	_____	_____
Flowrate:	_____tcfm	_____tcfm

Average Flowrate During Sampling:_____tcfm

Date and Time Cartridge Installed:_____ A.M. P.M. ___/___/___

Date and Time Sampling Started: _____ A.M. P.M. ___/___/___

Date and Time Sampling Stopped: _____ A.M. P.M. ___/___/___

Date and Time Cartridge Removed: _____ A.M. P.M. ___/___/___

PS-1 Sampler IEPA Number:_____

Sample Cartridge Number:_____ Blank Cartridge Number:_____

Comments:_____

_____

For Laboratory Use

_____

_____

_____

_____

_____

ILLINOIS ENVIRONMENTAL PROTECTION AGENCY

SAMPLE NO. _____

CARTRIDGE NO. _____ Collector _____

COMMENT _____

CHAIN OF CUSTODY RECORD

DATE	TIME	ACTION TAKEN AND SIGNATURE

APPENDIX G

Work to be Performed Portion of the Illinois EPA/Radian Corporation Contract for PCB Analysis

Work to be Performed --

General

The Agency will collect a total of 16 air samples at two locations in the southeast part of Chicago, Illinois. The samples will be taken at a nominal rate of two per week dependent upon meteorological and emission source conditions. The collected samples will be shipped to the contractor for analysis for polylchlorinated biphenyls (PCBs).

Cartridge Preparation

The contractor shall perform an initial cleanup of ten glass sampling cartridges. These sampling cartridges will be used interchangeably in the sample collection phase of the project. At the beginning of the contract, the Agency will ship (at its expense) the ten cartridges, each containing Florosil solvent sandwiched between two polyurethane foam plugs. Details of the initial cleanup are contained in Attachment A, Sections 1, 4, and 5.

The contractor shall perform an analysis of six clean (blank) glass fiber filters from those supplied by the Agency. The contractor will individually package 32 new glass fiber filters for field use. See Attachment A, Sections 2, 4, and 7.

After sample collection and analysis, the contractor will prepare the cartridges for the next sample event. Counting the ten cartridges from the initial cleanup, a total of 32 prepared cartridges (16 field samples, 16 field blank) will be used in the project. All shipment to and from the field site will be at Agency expense. See Attachment A, Sections 3, 9, and 12.

Sample Module Tests

The contractor will perform a test to determine the recovery efficiency of two PCB spiked sampling cartridges. After these tests are completed, a cleanup test of the spiked cartridges will be completed. Details are presented in Attachment A, Section 6.

Sample Analysis

The Agency wll ship the sampling cartridge and field blank to the contractor after field use. The contractor shall analyze the field cartridges for PCBs according to Appendix A, Section 3. The field blank will be extracted but will only be analyzed if the sample has a PCB value greater than the minimum detectable. See Attachment A, Sections 3 and 11 and Attachment B, Item 3.

Reports and Documentation

The contractor will provide biweekly verbal reports to the Agency. No later than 60 days after the last sample is sent to the contractor, a written report containing the data and any necessary explanations will be submitted to the Agency. The contractor will also submit all pertinent procedures and methods at the end of the project. See Attachment A, Section 10.

Contractor Consultation

The contractor shall provide consultation services to the Agency to assist in the preparation of a PCB quality assurance program. See Attachment A, Section 8.

Attachment A

SUMMARY OF PCB ANALYTICAL PROTOCOL

1. (a) The contractor shall perform an initial cleanup of ten glass sampling cartridges containing Florosil sorbent sandwiched between two polyurethane foam (PUF) plugs. Each sampling cartridge will be placed in a 500 ml Soxhlet and extracted with 200-300 ml of acetone for 14-24 hours at approximately 4 cycles per hour. The extracts will be discarded and the cartridges dried in a vacuum oven at room temperature.

   (b) Of the ten cartridges cleaned in Part 1a two cartridges will be re-extracted in 500 ml Soxhlets with 200-300 ml 5% diethyl ether in n-hexane (v/v) for 14-24 hours at approximately 4 cycles per hour. The extracts will be concentrated as per Part 4 and analyzed as per Part 5. Neither cartridge may contain more than 50 nanograms of PCB as Aroclor 1242. If either cartridge exceeds this limit, then Part 1a and 1b must be repeated until the limit is met.

   (c) The cleaned cartridges will be wrapped in n-hexane rinsed aluminum foil and placed in clean containers. These containers will be stored in a clean area until needed for field use.

2. The contractor shall perform an initial blank analysis of 6 randomly selected glass fiber filters from among those supplied by the IEPA. the 6 filters will be grouped together in one Soxhlet and extracted with 5% diethyl ether in n-hexane (v/v) for 14-24 hours at approximately 4 cycles per hour. After extraction the filters are discarded. The extract is then concentrated and analyzed as presented in Parts 4 and 5. The results of this analysis will be reported in micrograms PCB per filter.

3. (a) The contractor will individually extract 16 sample cartridges with the corresponding glass fiber filter samples. The contractor will also individually extract 16 trip blank cartridges with the corresponding filter trip blanks. Each cartridge-glass fiber filter pair is extracted in a 500 ml Soxhlet with 200-300 ml of 5% diethyl ether in n-hexane (v/v) for 14-24 hours at approximately 4 cycles per hour. Only the extract from each sample cartridge-filter pair will be initially concentrated and analyzed per Parts 4 and 5. If PCBs are detected then the corresponding extract from the trip blank cartridge-filter pair will be concentrated and analyzed per Parts 4 and 5. If no PCBs are measured from the sample extract, then the blank extract is discarded.

   (b) Following extraction the filters are discarded and the cartridges dried in a vacuum oven at room temperature. The dried cartridges are then wrapped in n-hexane rinsed aluminum foil and placed in clean containers. These containers will be stored in a clean area until needed for field use.

Page 2

4. Soxhlet extracts from Parts 1a, 2 and 3a will be individually concentrated to 5-10 ml in Kuderna-Danish evaporators. A few drops of mercury will be added to the extract to remove sulfur contaminants and the contents are agitated. The solution is then decanted, with n-hexane rinsings, into a glass vial to remove the mercury and reduced to approximately 1 ml under a stream of nitrogen gas. This concentrated solution is added to the top of a 10 cm, activity grade IV, alumina filled column, along with subsequent n-hexane rinsings of the glass vial. The column is eluted with 10 ml n-hexane. The eluate is collected and reduced to approximately .5 ml under a stream of nitrogen gas.

   NOTE: Section 11 of the USEPA Method 608 may be used instead of the procedure outlined in Part 4. However, final volumes must be approximately .5 ml.

5. Analysis of the concentrates will be performed using a gas-liquid chromatograph (GC) equipped with an electron capture detector. Instrument parameters shall be consistent with USEPA Method 608. The GC's lowest detection limit must be 500 picograms or less. Injection volumes must be between 5 and 10 microliters. Sample and trip blank results will be reported in micrograms PCB as Aroclor 1242 per cartridge-filter pair.

6. (a) Prior to field sampling the contractor will conduct a spike recovery test at two different PCB levels. To one clean sampling cartridge .12 ug of Aroclor 1242 will be injected just below the upper PUF surface. To another clean sampling cartridge .24 ug of the Aroclor will be added. Each cartridge will be individually extracted as presented in Part 3, concentrated as per Part 4, and analyzed in duplicate as per Part 5. Results will be expressed in micrograms PCB recovered.

   (b) The contractor will also perform a clean-up test on the cartridges used in Part 6a. After the Part 6a extraction, the two cartridges will be immediately re-extracted, concentrated, and analyzed in duplicate as per Parts 3, 4, and 5. The results are reported in micrograms PCB carried over.

7. The contractor will individually package 32 new glass fiber filters in sealed n-hexane rinsed aluminum foil pouches.

8. The contractor will provide 6 hours of verbal analytical expertise in assisting the IEPA to develop a PCB QA program plan.

9. The contractor will clean and reclean all containers before each field trip.

Page 3

10. (a) At the end of the project all standard procedures and methods, as well as quality control and quality assurance, will be documented and submitted to the manager of the Ambient Air Monitoring Section, Division of Air Pollution Control. All PCB data obtained during the course of the project will also be documented and submitted with the procedures.

    (b) During the project, biweekly reports will be verbally obtained.

11. All sample and trip blank concentrates from Part 4 will be stored at 4°C until the end of the project. At that time, these solutions may be shipped to the IEPA upon request.

12. The IEPA will provide a means for shipping all sampling materials between the contractor and the IEPA. Shipping costs will be paid by the IEPA.

Attachment B

SCHEDULE OF LABORATORY SERVICES AND COSTS

Item	Cost
1. Extraction of sixteen samples and sixteen field blanks, initial clean-up of ten cartridges, two spike recovery and clean-up tests, analysis of blank glass fiber filters, packaging of 32 filters, clean-up of cartridge containers, and 3 hours consulting to assist development of a QA program plan.	$4970
2. Concentration and analysis of 16 samples.	$2160
3. Concentration and analysis of trip blanks and additional samples.	$50/sample
4. Biweekly reports.	$420
5. Preparation and submittal of procedures and data	$2000

Total cost: $9550 + $50/sample analysis in excess of 16

DK/ct,dks,jd,bjh/sp/1729D/1-69,sp

50272-101

REPORT DOCUMENTATION PAGE	1. REPORT NO. IEPA/APC/85-003	2.	3. Recipient's Accession No. PB85 178424/AS
4. Title and Subtitle: Southeast Chicago Air Quality: A Plan for the Evaluation of PCB's			5. Report Date: February 1985(Approval)
			6.
7. Author(s): Ambient Air Monitoring Section			8. Performing Organization Rept. No. IEPA/APC/85-003
9. Performing Organization Name and Address: Illinois Environmental Protection Agency, Division of Air Pollution Control, 2200 Churchill Road, Springfield, IL 62706			10. Project/Task/Work Unit No.
			11. Contract(C) or Grant(G) No. (C) (G)
12. Sponsoring Organization Name and Address: Illinois Environmental Protection Agency, Division of Air Pollution Control, 2200 Churchill Road, Springfield, IL 62706			13. Type of Report & Period Covered: Final
			14.

15. Supplementary Notes

16. Abstract (Limit: 200 words)

A two station polychlorinated biphenyl (PCB) air sampling and analysis program is described in this report. The program was designed to measure PCB's in the vicinity of a hazardous waste incinerator that burned liquid and solid waste containing PCB's. The objectives of the sampling program, the sampling and analysis techniques, and the quality assurance procedures are presented.

17. Document Analysis a. Descriptors

polychlorinated biphenyls
air pollution sampling
incinerators
hazardous materials

b. Identifiers/Open-Ended Terms

PCB's
Chicago, Illinois

c. COSATI Field/Group

18. Availability Statement: Available from National Technical Information Service, Springfield, VA 22161	19. Security Class (This Report): Unclassified	21. No. of Pages
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(See ANSI-Z39.18) See Instructions on Reverse OPTIONAL FORM 272 (4-77) (Formerly NTIS-35) Department of Commerce